

The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

Volume 67 Number 8

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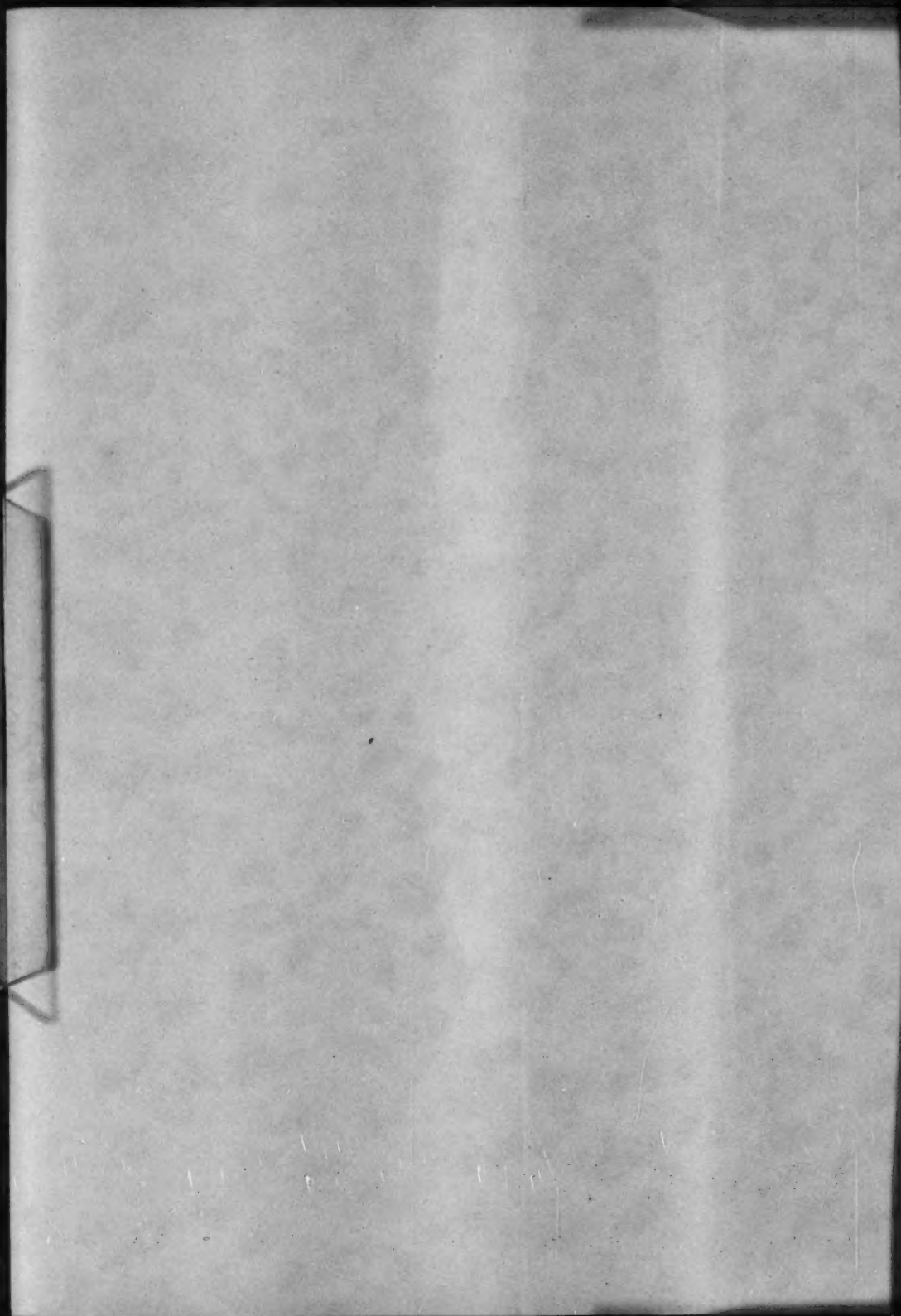
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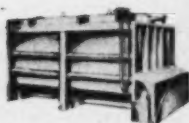
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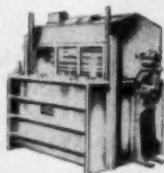
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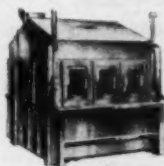
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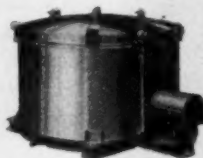
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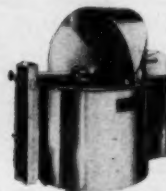
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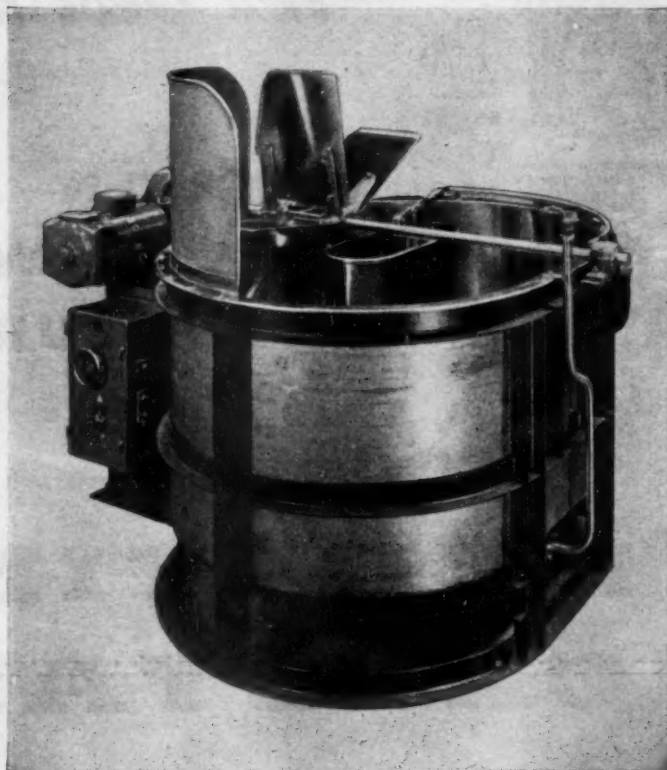
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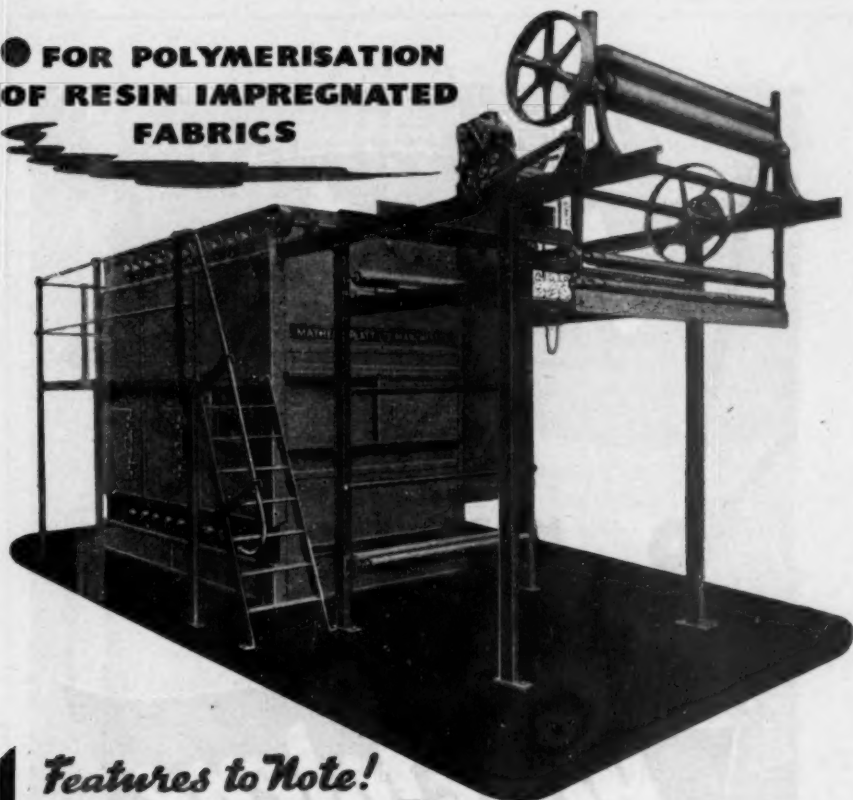
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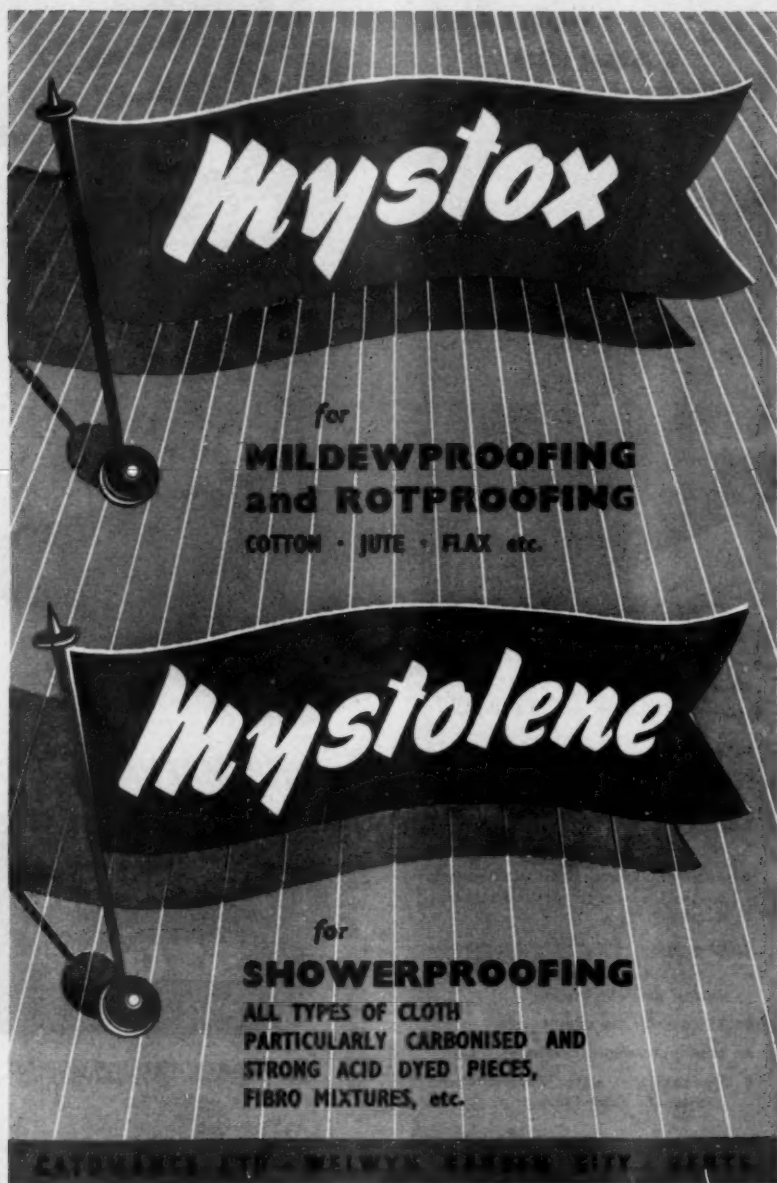
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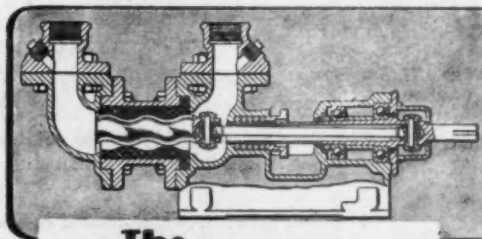
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Readers requiring general information regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1951 and pages 253-256 of the July 1951 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, 32-34 Piccadilly, Bradford, Yorkshire (Telephone Bradford 25138-9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

- | | |
|---|------------------------|
| Rayon Staple— A Dyeing Miscellany | <i>J. Boulton</i> |
| Causes of Uneven Dyeing of Cotton Threads | <i>I. Gailey</i> |
| Recent Developments in Fluorescent Lamps, with Particular Regard to Colour Problems | <i>S. T. Henderson</i> |
| The Dyeing of Silk and Silk Mixture Materials | <i>A. Thomson</i> |

COMMUNICATIONS

- | | |
|---|---------------------------------------|
| Advances in the Application of Vat Dyes to Viscose Rayon Cakes | <i>H. Hampson</i> |
| The Preparation and Properties of Regenerated Cellulose containing Vinyl Polymers. I— Internal Deposition of Polymers | <i>G. Landells and C. S. Whewell</i> |
| The Colorimetric Determination of Indigo | <i>J. Lotichius and J. Kooyman</i> |
| The Removal of Impurities from Grey Cotton. I— A Review of Developments in Plant for Alkali Boiling | <i>H. A. Turner and O. J. Hvattum</i> |



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THE JOURNAL OF THE Society of Dyers and Colourists

Volume 67—Number 8

AUGUST 1951

Issued Monthly

COMMUNICATIONS

Reduction of Direct Dyes during the Dyeing of Viscose Rayon

W. ARMFIELD

The cause of reduction of certain direct dyes during the dyeing of viscose rayon at temperatures near the boil is the presence of alkali, and some direct dyes contain sufficient alkali to cause reduction of other direct dyes although they themselves may not be affected.

With increase in temperature reduction increases, and at the boil considerably more reduction takes place than at 90°C.

Dyeing in enclosed machines favours reduction owing to the restricted air space, but the effect of this can be overcome by blowing air over the surface, or through the dye liquor. Lowering the pH of the dye liquor decreases the tendency of dyes to reduce during dyeing. Addition of such a substance as ammonium sulphate or chloride prevents reduction altogether even when dyeing at temperatures near the boil, in all but exceptional cases.

The pH values of a number of aqueous dye solutions are listed, and a simple method is described whereby the susceptibility of direct dyes to reduction can be determined. A number of dyes have been tested, and are classified into four groups, according to whether they show considerable, moderate, slight, or no reduction.

Introduction

From time to time it has been noticed in the Droylsden dyehouse that certain direct dyes reduce during the course of dyeing of viscose rayon cakes. Reduction of dyes during skein dyeing on Gerbert-type machines has also been noticed on rare occasions. In order to save fuel by reducing heat losses, some of the cake-dyeing machines have been enclosed, and on these machines reduction of dyes has occurred to a greater extent than on the uncovered machines. Increased reduction trouble has also been reported in the case of enclosed winches, compared with the ordinary open winch¹.

Some dyes appear to be more susceptible to reduction than others, but a dye which reduces on one occasion may not do so on another, and the extent to which dyes reduce varies considerably under what are apparently similar conditions of dyeing. Dye reduction may occur and produce different effects according to the particular dye, some dyes on reduction changing hue completely—e.g. a direct blue may turn yellow—and others altering in hue without losing much in depth, but if the reduction is severe enough the dye in some cases becomes completely decolorised.

Cellulose in hot alkaline solution forms a reducing system sufficiently strong to cause the reduction of direct dyes², which would appear to indicate that alkali in some form must be present in the normal direct-dye dyeing process. Since the normal dyeing process involves the use of yarn, water, dye, sodium chloride, and sometimes dyeing assistant, one or more of these must be the source of alkali. Soap used for dyeing purposes is appreciably alkaline, and obviously should not be used in a dye

mixing containing dyes which reduce easily. The dyes which are most easily reduced are the drabbing dyes such as blues, greys, blacks, and these appear to reduce more easily when present in the dye solution in low concentrations.

As a result of these works observations, laboratory experiments were carried out in order to find (i) the cause of this reduction trouble in the dyehouse; (ii) a means of preventing this reduction, and (iii) a means of testing dyes for susceptibility to reduction.

Experimental

One recipe which has given considerable reduction trouble during the dyeing of cakes consisted of a mixing of—

1%	...	Diazol Light Yellow N4J
0.5%	...	Durazol Blue G 200
10%	...	Sodium chloride
0.025%	...	Calgon T (Albright & Wilson)

Dyeing had been carried out at the boil in 12 volumes using Manchester Corporation water. The blue dye after a time gradually disappeared from the solution and the yarn, leaving the latter a flat yellow in appearance instead of a bright green.

Using the above recipe, a laboratory experiment was done at the boil under reflux in Pyrex glass apparatus and with 150/27 viscose rayon. The same effect as in the dyehouse was obtained, the blue dye gradually disappearing from the yarn and the solution, until after two hours the yarn became bright yellow, corresponding to a self shade of Diazol Light Yellow N4J.

The experiment was repeated, dyeing for two hours without sodium chloride or Calgon T, and

again the blue dye disappeared entirely. Then the original recipe was used in distilled water, but with exactly the same result. The experiment was repeated using Durazol Blue G 200, sodium chloride, and Calgon T in Manchester Corporation water, but omitting the yellow dye component. A blue dyeing was obtained which appeared to be of the correct depth and hue. This indicated that Diazol Light Yellow N4J must have been responsible in some way for the reduction trouble in this mixing. An examination of the latter dye showed that it contained 42% sodium carbonate, and on making up a solution of Diazol Light Yellow N4J in distilled water so as to give a solution equivalent to 1% dye on the weight of yarn in 12 volumes, the pH was found to be 10.23. The effect of dyeing with 1% Diazol Light Yellow N4J, therefore, is to introduce 0.42% sodium carbonate based on the weight of yarn into the dye solution. 150/27 Viscose rayon was next dyed with 0.5% Durazol Blue G 200, sodium chloride, Calgon T, and Manchester Corporation water at the boil under reflux, with the addition of 0.42% sodium carbonate based on the weight of yarn: the dye was completely reduced in less than two hours' dyeing.

It has thus been established that for the particular mixing under consideration the source of alkali is Diazol Light Yellow N4J, and that the alkali is sufficient to cause complete reduction of Durazol Blue G 200 in presence of viscose rayon at the boil.

In view of the excessive alkali content of Diazol Light Yellow N4J, the pH values of a number of commercial direct dyes were determined. The dye solutions were made up in distilled water so as to give the equivalent of a 1% dyeing of standard-brand dye in 30 volumes. This latter volume was chosen as an average, reduction having sometimes occurred in practice in larger volumes than 30. The pH values shown in Table I were determined at 20°C. using a glass electrode.

It will be seen from Table I that there is an appreciable variation in pH between the dyes tested, and a few dyes are noticeably alkaline. It is also obvious that a dye which is reduced in bulk dyeing in some instances may not be so in others, depending on the dyes in the mixing.

The effect of the yarn had not so far been considered, and therefore the following experiment was done on 150/27 commercial-production viscose rayon washed twice with distilled water at 60°C. for 20 min. and dried at 70°C. Washed and unwashed yarns were dyed with the Diazol Light Yellow N4J-Durazol Blue G 200 mixing mentioned previously, with 10% sodium chloride at the boil under reflux for 2 hr. in 12 vol. At the same time a blank was carried out, i.e. a similar experiment with the yarn omitted. After two hours no reduction of dye occurred in the case of the blank, but the washed and unwashed yarns showed reduction of dye to the same extent. The dyeings were inspected frequently during the course of dyeing, but the washed and unwashed yarns and their dye liquors showed the same rate of reduction. This experiment indicates that

there is no extraneous matter in the normal viscose rayon production liable to cause dye reduction, but that reduction of direct dyes can take place in presence of viscose rayon and small concentrations of alkali. As stated previously, the sodium carbonate from the Diazol Light Yellow N4J corresponds to 0.42% on the weight of yarn, which means that in 12 volumes the concentration of the solution is only 0.035%.

Experiments were next carried out to determine the effect of variations in pH, temperature, and amount of oxygen, the latter on account of the noticeably greater number of cases of reduction in bulk practice with enclosed machines compared

Strength (%)		pH
1.0	Diazol Light Yellow N4J (Fran) ...	10.13
1.0	Diamine Fast Yellow 4G (IG) ...	9.96
1.0	Diazol Light Yellow N2J (Fran) ...	9.62
1.0	Viscose Blue Grey NB (Fran) ...	9.21
1.0	Diazo Brilliant Orange GR (IG) ...	9.20
0.67	Chlorantine Fast Green 5GLL 150 (CAC) ...	9.12
0.33	Chloramine Black BH 300 (S) ...	8.92
1.0	Viscose Navy Blue NB (Fran) ...	8.82
0.67	Chlorazol Dark Green PL 150 (ICI) ...	8.55
0.17	Chlorazol Blue B 600 (ICI) ...	8.49
0.32	Chlorazol Black E 310 (ICI) ...	8.24
0.67	Durazol Orange 4R 150 (ICI) ...	8.16
1.0	Solophenyl Red Brown (Gy) ...	7.80
1.0	Diphenyl Fast Rubine RL (Gy) ...	7.72
1.0	Diphenyl Fast Blue 3RL (Gy) ...	7.64
0.5	Durazol Blue 2GN 200 (ICI) ...	7.57
1.0	Chlorantine Fast Green BLL (CAC) ...	7.44
0.5	Chlorazol Fast Helio 2RK 200 (ICI) ...	7.35
0.56	Durazol Grey RG180 (ICI) ...	7.11
0.44	Pyrazol Orange GH 225 (S) ...	7.11
1.0	Icyl Orange RS (ICI) ...	7.10
0.8	Durazol Orange 2G 125 (ICI) ...	7.08
1.0	Diphenyl Fast Blue 10 GL (Gy) ...	7.02
1.0	Durazol Orange Brown 2 RS (ICI) ...	7.01
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0.8	Chlorazol Fast Scarlet 8B 125 (ICI) ...	6.98
0.67	Durazol Scarlet 2G 150 (ICI) ...	6.97
1.0	Durazol Violet 2BS (ICI) ...	6.94
1.0	Benzanil Fast Bordeaux 2 BLN (YDC) ...	6.90
1.0	Diphenyl Fast Blue Green BL (Gy) ...	6.90
1.0	Benzopurpurine 10 B (S) ...	6.89
1.0	Chlorantine Fast Blue 3GLL (CAC) ...	6.87
0.5	Chlorazol Fast Pink BK 200 (ICI) ...	6.83
0.33	Diphenyl Fast Yellow FF 300 (Gy) ...	6.79
1.0	Solar Orange RGL (S) ...	6.73
0.5	Durazol Blue 2R 200 (ICI) ...	6.72
0.67	Chlorazol Blue G 150 (ICI) ...	6.70
1.0	Diphenyl Brilliant Violet 3B (Gy) ...	6.68
0.56	Diphenyl Fast Red 7BL 180 (Gy) ...	6.66
1.0	Direct Blue 5B (Fran) ...	6.64
0.5	Durazol Blue G 200 (ICI) ...	6.60
0.5	Chlorantine Fast Blue GLL 200 (CAC) ...	6.57
0.22	Rigan Sky Blue G 450 (CAC) ...	6.40
0.56	Solar Yellow 2R 180 (S) ...	6.05
1.0	Benzo Fast Light Scarlet 4BL (IG) ...	5.43

with open machines. In all the following experiments, unless otherwise stated, the yarn used was 150/27 viscose rayon washed twice with distilled water at 60°C. for 20 min. and dried, then dyed in 12 vol. for 2 hr.

EFFECT OF OXYGEN

The following mixing was dyed under reflux at the boil—

1.0%	...	Diazol Light Yellow N4J
0.5%	...	Durazol Blue G 200
10%	...	Sodium chloride

A leading tube extending to the bottom of the condenser introduced (a) air, (b) oxygen, or (c) nitrogen at 400 c.c./min. A leading tube was also used in another set of experiments to bubble these three gases through the dye solutions at the same rate as above.

With nitrogen complete reduction of the blue dye was obtained, with air the reduction was considerably decreased, with oxygen a further slight improvement was obtained. There appeared to be no advantage in actually bubbling air or oxygen through the dye liquor compared with blowing it on to the surface.

Dyeings were next carried out at the boil in Pyrex beakers using the same blue-yellow mixing and quantity of sodium chloride. The beakers were (i) uncovered, (ii) covered with a clock glass, and (iii) covered by suspending a clock glass at a distance of 0.5 in. above the top of the beaker. Frequent additions of boiling distilled water were made to (i) and (iii) to make up the losses due to evaporation and so keep the volume of dye liquor constant.

With (ii) complete reduction of the blue dye occurred, with (iii) moderate reduction, and with (i) no reduction. It is demonstrated quite clearly, therefore, that accessibility of oxygen to the dye liquor surface greatly decreases the tendency of dyes to be reduced.

EFFECT OF TEMPERATURE

The same blue-yellow mixing with 10% sodium chloride was used under reflux at the boil, at 95°C., and at 90°C. The blue dye was completely reduced at the boil, very slightly reduced at 95°C., and not at all at 90°C.

EFFECT OF pH

Again using the same blue-yellow mixing with 10% sodium chloride at the boil under reflux, dyeings were carried out at pH 9.0, 8.5, 8.0, 7.5, and 7.0, the pH of the solutions being adjusted with hydrochloric acid. With decrease in pH the degree of reduction of the blue dye also decreased, but at pH 7.0 some reduction was still obtained. After dyeing and cooling, the pH of the dye liquor originally at 7.0 was found to be 8.7. Since the original pH measurements were made at 20°C., all the carbon dioxide generated by the action of hydrochloric acid on the sodium carbonate in the dye would not have been driven from the dye liquor, but it would all be driven off during boiling, so that the pH would rise. Nevertheless, it has been demonstrated that on lowering the pH, the degree of reduction is decreased.

A neutralisation process of the above type is not suitable for works practice, since it would be difficult to control, but Ashpole *et al.*¹ have shown that additions to the dye liquor of potentially acid

substances such as ammonium sulphate will prevent reduction of direct dyes during the dyeing process.

CLASSIFICATION OF DIRECT DYES

Experience had shown that some direct dyes reduce more easily than others under practical dyeing conditions, so experiments were carried out in order to try to find a suitable method for the classification of direct dyes, according to their susceptibility to reduction. Since borax is a mild and stable alkali, it was decided to use this substance as the alkali in controlling the reducing process. The dyeing temperature selected was 98°C. instead of at the boil, so as to avoid any possible variations in rate of boiling. Dyeings with 1% of standard-brand dye or its equivalent were done on 150/27 viscose rayon, washed twice at 60°C. as previously described, with the addition of 5% sodium chloride on the weight of yarn, at 98°C. in 12 vol. for 2 hr. with additions of (a) 0, 0.02, 0.04, 0.06, and 0.10% solution of borax. The vessels used for dyeing were 250-c.c. Pyrex conical flasks fitted with rubber stoppers carrying glass capillary tubes. The yarn, in the form of 15-g. skeins, was suspended in the dye liquor by means of a wire passing through the capillary tube and attached at the other end to a multiple stirring device, such that all the dyeings for the full borax range could be carried out at the same time. The flasks were immersed in a thermostatically controlled oil-bath so as to maintain a dyebath temperature of $98 \pm 0.25^\circ\text{C}$. The stirring device not only kept the yarn in motion, but also served to maintain an even temperature throughout the dye liquor.

A number of direct dyes, chiefly those which have been found to reduce in practice, were dyed according to the above procedure. It was found that some of the dyes showed some reduction with 0.02% borax solution and others no reduction even with 0.1%. It was therefore possible to arrange the dyes in groups showing, according to visual assessment, considerable, moderate, slight, and no reduction respectively. Standard dyeings for comparison were done with 5% sodium chloride at 90°C. for 2 hr. in 12 vol. in unstoppered flasks.

Reduction was indicated by loss in depth, usually accompanied by change in hue. There was no difficulty in telling whether loss of depth was due to reduction: comparison with the borax range of dyeings was sufficient indication, since they were all done at the same temperature. Table II shows

TABLE II Dye	
Strength (%)	CONSIDERABLE REDUCTION (Begins at > 0.02% borax soln.)
0.22	Rigan Sky Blue G 450 (CAC)
0.4	Diphenyl Fast Blue Green BL 250 (Gy)
0.5	Chlorantine Fast Blue GLL 200 (CAC)
1.0	Diphenyl Fast Blue 3RL (Gy)
0.5	Durazol Blue 2R 200 (ICI)
0.5	Durazol Blue 4R 200 (ICI)
0.5	Durazol Blue G 200 (ICI)
1.0	Diphenyl Fast Blue 10 GL (Gy)
0.5	Chlorantine Fast Blue 3GLL 200 (CAC)
0.5	Chlorantine Fast Green 5GLL 200 (CAC)
1.0	Chlorantine Fast Green BLL (CAC)

MODERATE REDUCTION
(Begins at $\geq 0.04\%$ borax soln.)

- 1-0 Durazol Red 6BS (ICI)
- 1-0 Icyl Blue 2RS (ICI)
- 0-48 Solar Orange RGL 210 (S)
- 1-0 Icyl Blue G (ICI)
- 1-0 Diphenyl Green 2G (Gy)
- 1-0 Rigan Blue R (CAC)
- 0-67 Chlorazol Blue G 150 (ICI)
- 1-0 Durazol Grey RG (ICI)

SLIGHT REDUCTION
(Begins at $> 0.06\%$ borax soln.)

- 0-67 Diphenyl Fast Red 7BL 150 (Gy)
- 0-33 Diphenyl Brilliant Blue FF 300 (Gy)
- 0-67 Durazol Grey VG 150 (ICI)
- 0-33 Chloramine Black BH 300 (S)

NO REDUCTION
(Begins at $> 0.06\%$ borax soln.)

- 0-6 Chlorazol Dark Green PL 165 (ICI)
- 1-0 Chlorazol Fast Orange G (ICI)
- 0-67 Durazol Orange 2G 150 (ICI)
- 0-67 Durazol Rubine B 150 (ICI)
- 0-5 Pyrazol Orange GH 200 (S)
- 1-0 Diazo Brilliant Orange GR (IG)

a list of dyes tested and classified according to their susceptibility to reduction, each class being indicated by a certain maximum borax concentration at which reduction begins.

Thus if the first appearance of reduction of a dye under test occurs between 0 and 0.02% borax solution, it should be classified as susceptible to considerable reduction; if reduction begins at 0.02-0.04% borax solution, it should be classified as susceptible to moderate reduction; and so on. From the dyes tested three were selected as standards, so that comparative dyeings could be carried out if desired to compare with tests on dyes not appearing in Table II. Chlorantine Fast Green BLL can be regarded as the standard or upper-limit dye for the "considerable reduction" class, Durazol Grey RG for the "moderate reduction" class, and Chloramine Black BH for the "slight reduction" class.

In Table II the dyes are arranged in order, so that Rigan Sky Blue G 450 is the most easily reduced and Chloramine Black BH the least easily reduced of those which do actually show reduction under the test conditions. Although Rigan Sky Blue G 450 and Chlorantine Fast Green BLL are in the same group with regard to the commencement of reduction, viz. 0.0-0.02% borax solution, the former dye is much more severely reduced. It was thought advisable, therefore, to carry out some experiments using Rigan Sky Blue G 450 in order to find the effect of pH and temperature on a dye which is very easily reduced and to see how reduction of such a dye could be prevented in practice.

In order to try the effect of ammonium sulphate on a very easily reduced dye in presence of a dye containing excessive alkali, the following experiments were carried out.

Dyeings were done with—

- 1% Diazol Light Yellow N4J
- 0.22% Rigan Sky Blue G 450
- 5% Sodium chloride

in 12 volumes for 2 hr. at 98°C. on washed 150/27 viscose rayon yarn. In this and all subsequent experiments the yarn was dyed in conical flasks fitted with the stirring device described previously. Additions of 0, 0.4, 0.6, 0.8, and 1.0% ammonium sulphate were made to the dye solutions before entering the yarn. With 1.0% ammonium sulphate no reduction occurred, with 0.8% very slight reduction, with 0.6% and 0.4% considerable reduction, and without any addition of ammonium sulphate the blue dye was completely reduced.

The above experiment was repeated at 90°C. With 0.4% ammonium sulphate or more no reduction occurred, but without any addition the blue dye was again completely reduced. In the case of Durazol Blue G 200 it has been shown previously that no reduction was obtained at 95°C, even without ammonium sulphate. With Rigan Sky Blue G 450, even at 90°C. ammonium sulphate is necessary to prevent reduction with Diazol Light Yellow N4J in the dyebath, although the concentration of ammonium sulphate required is much less than at 98°C.

TABLE III

		pH at 20°C.		Remarks
		Before Dyeing	After Dyeing	
(1)	No addition ...	10.43	8.73	Blue dye completely reduced
(2)	pH adjusted with— Hydrochloric acid ...	7.0	8.72	Blue dye considerably reduced
(3)	Sulphuric acid ...	7.0	8.63	Less reduction than in (2)
(4)	Acetic acid ...	7.0	8.82	As in (2)
(5)	1% Ammonium sulphate	9.04	9.20	No reduction

Further dyeings were carried out with the above mixing under the same conditions but with the additions shown in Table III, from which it is quite obvious that to determine the pH of the dye solution cold and thereby to assess its tendency to reduction may be entirely misleading. The pH alone is obviously not a sufficient indication as to whether dyes will reduce during dyeing, and, as shown previously, there is no evidence that decreasing the initial alkalinity of the dye liquor diminishes the tendency for dyes to reduce. Such substances as ammonium sulphate are effective because at higher temperatures they dissociate to a greater extent and become acidic in character.

In a dye mixing in which one dye is responsible for the reduction of another, the proportion and concentration of each dye will affect the rate and degree of reduction. The following experiment was done in order to find how much ammonium sulphate would be required to prevent reduction of Rigan Sky Blue G 450 in presence of a relatively large concentration of Diazol Light Yellow N4J such as might occur in practice. Washed yarn as before was dyed with—

- 5% Diazol Light Yellow N4J
- 0.22% Rigan Sky Blue G 450
- 10% Sodium chloride

in 12 vol. at 98°C. for 2 hr. with additions of 0, 0.5, 1.0, 2.0, 5.0, and 10.0% ammonium sulphate.

Complete reduction of the dye occurred up to and including 2% ammonium sulphate; 5% and 10%

gave dyeings very much alike but still showing appreciable reduction. The experiment was repeated at 90°C. when 10% ammonium sulphate showed no reduction, 5% very slight reduction, 2% moderate reduction, and the lower amounts complete reduction of Rigan Sky Blue G 450.

Discussion of Results

Reduction of direct dyes during the dyeing of viscose rayon in bulk practice has been shown to be due to the presence of alkali, and some direct dyes contain alkali in sufficient quantity to cause reduction of other dyes. Reduction which occurs readily at the boil is decreased by lowering the dyeing temperature, but the disadvantage of dyeing below the boil is that the rate of diffusion of the dye into the fibre is decreased. This means that in practice a longer time is required in which to level any initial unevenness in dyeing, such as a bad strike.

Restriction of the accessibility of air to the dye liquor surface also favours reduction, so that machines which have been specially enclosed, in order to economise in fuel, cause reduction trouble to a greater extent than the normal open machines. In the case of enclosed machines, air can be blown over the surface of the dye liquor or blown through it, to decrease any reduction tendencies.

The most satisfactory way of overcoming dye reduction appears to be control of the pH of the dye solution, and this can be done very conveniently by the addition of ammonium sulphate. The amount of ammonium sulphate required to prevent reduction varies according to the quantity of alkali present and also the time and temperature of dyeing.

No fixed amount of ammonium sulphate can be recommended to cover all possibilities, since this would entail the use of excessive amounts. Apart from being unnecessary in many cases, large amounts of ammonium sulphate would exert a considerable salting-on effect, which would cause uneven dyeing. In most cases 0.5% ammonium sulphate based on the weight of yarn should prevent reduction at temperatures very near the boil, but where this proves to be inefficient, further modification should be made, such as lowering the dyeing temperature. In extreme cases, however, even at 90°C. the amount of ammonium sulphate might have to be increased up to 5 or 10%.

In specially difficult cases, fuel economy might have to be sacrificed and the machine ventilated, or the temperature of dyeing lowered, necessitating longer levelling times.

By a combination of temperature adjustment, ventilation, and addition of ammonium sulphate, it should be possible to dye any mixing of direct

dyes on to viscose rayon without having reduction troubles.

Soap, because of its alkalinity, should not be used in the dye liquors where reducible dyes are concerned. Although the addition of ammonium sulphate would counteract soap alkalinity, the acidity developed by ammonium sulphate at temperatures near the boil is sufficient partly to crack the soap, and so trouble would occur in the form of soap marks. Soap is normally added to the dye liquor to maintain a clean solution and clean soft yarn, but it can be applied as a cold aftertreatment where ammonium sulphate has been added to the dye liquor. If desired, soap may be replaced by any of the neutral types of sulphated fatty alcohols etc.

Table II shows the considerable variation in reduction of direct dyes under identical dyeing conditions. With the dyes listed under considerable, moderate, and slight reduction, it would appear advisable to dye in presence of ammonium sulphate so as to avoid reduction. This is supported by practical experience, since dyeing at the boil in enclosed machines without ammonium sulphate has caused the partial reduction of such dyes as Diphenyl Fast Red 7BL and Durazol Grey VG, both of which are in the "slight reduction" class.

It should not be overlooked that, instead of preventing the reduction of certain dyes by the methods outlined above, it might be better to substitute for them, where possible, dyes which are free from trouble in this respect, and this also applies to dyes having an excessive alkali content, which are therefore liable to cause reduction of other dyes.

Dyeing conditions vary from dyehouse to dyehouse, and so the cause of reduction and the extent to which it occurs will also vary, but the experiments described above should provide a satisfactory guide to enable the dyer to overcome his own reduction difficulties.

* * *

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COURTAULDS LTD.
DROYLSDEN LABORATORY
MANCHESTER

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The Colour of Organic Compounds

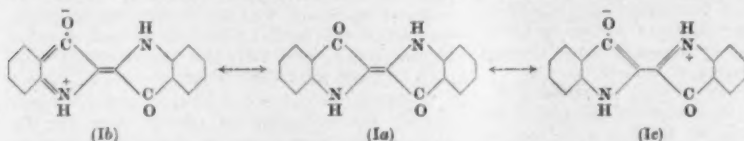
IV—Indigo and Related Dyes*

E. B. KNOTT

It is shown that the application to the indigo molecule of known colour rules, particularly the general rule given by the author¹, leads to the expectation of a deep colour. Various isomers, analogues, and derivatives of indigo and thioindigo are similarly examined, and their colour is explained on a qualitative basis.

INTRODUCTION

The problem of the deep colour of indigo has excited the attention of colour chemists for many years, mainly because a consideration of its structure with its short conjugated system leads one to expect a much lighter colour. In spite of the most ingenious attempts by various authors to justify this or that type of resonance as being the source of the deep colour, it is obvious that the solution to the problem is still lacking. It is now proposed to analyse the resonance system of indigo in a more detailed way than hitherto.



THE ENERGETIC SYMMETRY OF THE INDIGO MOLECULE†

Indigo exists as the planar *trans*-isomer (Ia)³, the *cis*-isomer being overcrowded and non-planar. There exists the possibility, therefore, of the presence in the molecule of two hydrogen bonds. It contains two well defined auxochromes of the $-M$ type (NH) and two of the $+M$ type (CO). The high intensity of the long-wavelength absorption band indicates that this is due to an $N \rightarrow V_1$ transition similar to that found in more simple dyes containing only two auxochromes.

It is generally recognised that the significance of the possible structures which contribute to the resonance hybrid of dyes depends largely upon their relative energies. In the case of indigo the low-energy extreme structure (Ia) contains two aromatic rings. Of the higher-energy dipolar extreme structures, Ic is one of two, in which

† A dye is energetically symmetrical when the energy levels of the extreme structures are identical. Such a dye is usually found only amongst ionic dyes, e.g. cyanines, in which two identical resonance structures may be drawn and in which the characteristic charge resides first on one then on the other auxochrome. Such a resonance system is quantum-mechanically degenerate. In non-ionic dyes such as indigo the resonance system may be "accidentally degenerate" in that it is possible that the energy of the classical extreme structure (e.g. Ia) may be exactly the same as that of the ionic extreme structure (e.g. Ic). By extreme structures are understood the two or more "low-energy" structures which can be drawn for all dyes. Excited or intermediate structures are those structures which can be visualised as contributing to the hybrid of a dye in the passage of an electron from one auxochrome to another. These are usually of higher energy than the extreme structures².

polarisation is along the length of the molecule and which was advocated by Arndt⁴ as being the most significant of this type. For its contribution charge separation is required, but full aromaticity is preserved in both benzene rings. Ib is one of three possible ionic extreme structures in which one or both rings are *o*-quinonoid and the polarisation is at right angles to the length of the molecule; the importance of these structures was stressed by Kuhn⁵ and supported by van Alphen⁶ and Gill and Stonehill⁷. However, for contributions by these *o*-quinones Kekulé resonance is lost in one or both

benzene rings, which together with the energy required for charge separation will make structures of type Ib of considerably

higher energy than either Ia or Ic. Although all these structures will contribute to the hybrid, the resonance system (Ia \leftrightarrow Ic) will be less asymmetric energetically (less non-degenerate) than (Ia \leftrightarrow Ib) and will therefore contribute more to the hybrid than the latter. It is moreover true⁸ that the colour of closely related dyes deepens as the energetic asymmetry of the resonance system decreases, and consequently the long-wavelength band of indigo must be attributed to the less asymmetric resonance system rather than to the more highly asymmetric one.

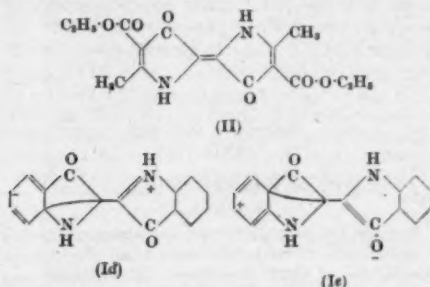
It may be assumed that Ic is of higher energy than Ia. However, the possible presence in the molecule of two hydrogen bonds⁹ will decrease this non-degeneracy, since these bonds will be stronger in the ionic structure (Ic), in which the electron density on the donor oxygen atoms is greater and the electrophilic nature of the acceptor hydrogen atoms is increased¹⁰. Indeed, this stabilisation of Ic appears to be sufficient to reduce the degree of non-degeneracy to a level such that the shifts of λ_{\max} of indigo in solvents of differing polarity are quite small. Thus it is known¹¹ that the sensitivity of λ_{\max} of non-ionic dyes to such changes in solvent polarity is at a minimum around the point of accidental degeneracy. It is believed, therefore, that one of the first requirements of deep colour, a near-degenerate resonance system, is present in the indigo molecule.

THE EFFECT OF THE BENZENE RINGS

van Alphen¹² has given the example of the dye (II), which is red, as an argument against the

* Communication No. 1330H from the Kodak Research Laboratories.

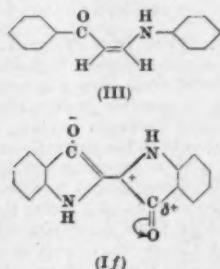
Arndt-type resonance, since in II the Kuhn-type resonance is not possible. It is well known, however, that the attachment of phenyl or benzo rings to the auxochromes of many dyes¹³ almost invariably results in a bathochromic shift, owing to



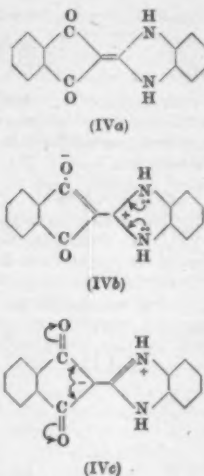
contributions by excited structures such as Id or Ie, in which a charge of either sign passes beyond the auxochromes into the benzene rings. This *extra-conjugation*, as it may be termed, is thus a second important contributor to the depth of colour of the dye.

RELATIVE ENERGY LEVELS OF EXTREME AND EXCITED STRUCTURES

It is known that in closely related dyes the colour will deepen as the energy level of any one significant excited structure increases with respect to the levels of the extreme structures¹. Indigo may be compared with the simpler β -anilinovinyl

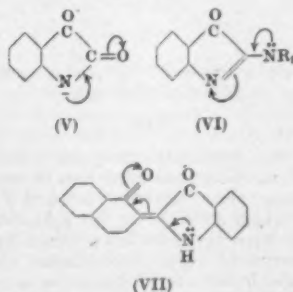


phenyl ketone (III), which is yellow ($\lambda_{\max} = 371 m\mu$ in methanol) and which contains the same Arndt-type resonance system and possibly one hydrogen bond. The excited structure If and the analogous structure from III may then be considered. The effect of the introduction of the second carbonyl group in passing from III to I will be to raise considerably the energy of the excited structure, resulting in If, for, in this structure, the carbonyl resonance indicated by a curved arrow will result in adjacent atoms of like charge. According to Pauling and Brockway¹⁴ contributions by such structures must be very small or negligible. In indigo, therefore, a significant redistribution of charge must occur when compared with III, and this redistribution according to the rule given above will result in a bathochromic shift. This, then, is considered to be the third significant factor.

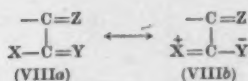


Van Alphen¹² synthesised the isomeric compound IVa, and because it is but pale yellow used it as an argument against the Arndt-type resonance. Structures IVb, c are important contributors to the hybrid, but in this case, in comparing IVa with III, the presence of the second -NH- group in IVb and of the second -CO- group in IVc will increase the significance of these two structures, since the latter are also important excited structures of the degenerate carboxyl-type ion in IVb and amidinium ion in IVc (curved arrows). The change from III to IVa, apart from considerations of any resultant change in energetic asymmetry, would therefore, on the basis of the above rule, work *hypsochromically*, as is the case.

Although the effect on colour of the presence in indigo of the crossed conjugation is not known, it is not believed to be of importance. This is shown by the exceedingly deep colours reported by van Alphen¹² of substances such as the anion of isatin (V) and VI, which are purple, and VII, which is blue, all of which contain but one -M auxochrome.



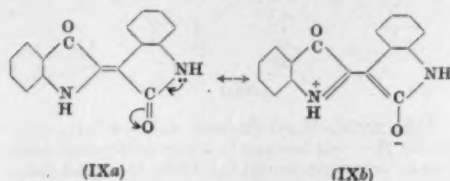
On the above basis it may be said that any compound containing the system VIII, where Y and Z are +M atoms or groups and X is a -M atom or group, will be "unexpectedly" deeply coloured assuming the resonance system VIIIa \longleftrightarrow VIIIb is



degenerate or near-degenerate. Apart from the examples quoted in this paper there are many more containing this system, particularly in the quinone and anthraquinone series.

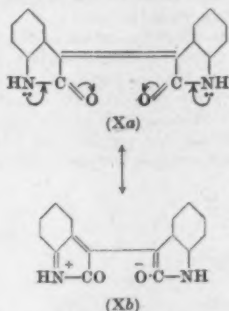
RELATED DYES

There are many dyes related to indigo which contain grouping VIII but which are not so deeply coloured because of the higher non-degeneracy of their resonance systems. The simplest analogue is thioindigo ($\lambda_{\max} = 545 \text{ m}\mu$ in xylene). In indirubin (IXa) the important extreme structures will be IXa, b. However, as the strong amide resonance (curved arrows in IXa), not involving loss of

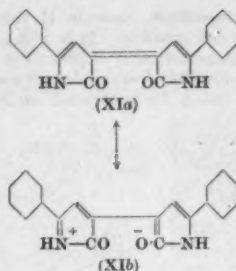


Kekulé resonance as in indigo, must be overcome before IXb can participate, the latter will be of considerably higher energy than IXa, so that an appreciable hypsochromic shift would be expected on proceeding from indigo to indirubin, as is the case.

In isoindigo (X), which is only yellow, Arndt-type resonance ($Xa \longleftrightarrow Xb$) involves the loss of aromaticity in one of the rings in Xb. For this

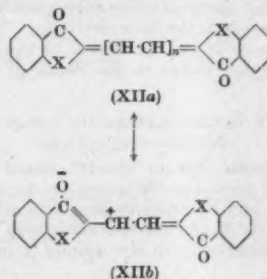


structure to contribute, the amide resonance shown by curved arrows must also be overcome. This system must therefore be highly asymmetric¹⁵, which accounts for the lightness of the colour. The hypsochromic effect of the interposition of the phenylene ring in the resonance system is made clearer in the above example by a consideration of dye XI¹⁶, a close analogue of X in which no loss of aromaticity is incurred on proceeding from XIa to XIb. In spite of the competitive amide resonance (cf. IX), this dye is magenta in pyridine ($\lambda_{\max} = 568 \text{ m}\mu$) and not dissimilar in colour from indirubin.



HIGHER VINYLOGUES OF INDIGO AND THIOINDIGO

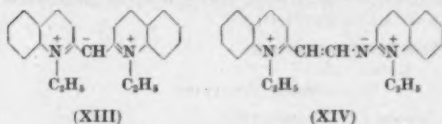
Perhaps nothing illustrates the abnormal colour of indigo and related dyes more than the absorption figures of their vinylogues. Friedländer and Risse¹⁷ found that the change from Ia to XIIa ($X = \text{NH}$, $n = 1$) produced a bathochromic shift



of only 5 mμ. in xylene, from 595 mμ. to 600 mμ. In normal dyes a considerable bathochromic shift would be expected from such a structural change. At first sight, a possible explanation might be found in the loss of hydrogen bonding, which would result in an increase in the energetic asymmetry of the molecule with a superimposed hypsochromic shift. This, however, cannot be the explanation, since the above authors also found that a similar structural change from thioindigo (XIIa, $X = \text{S}$, $n = 0$) to XIIa ($X = \text{S}$, $n = 1$) gave a hypsochromic shift from 545 mμ. to 508 mμ. On proceeding to XIIa ($X = \text{S}$, $n = 2$), λ_{\max} shifted bathochromically to 528 mμ., which falls short of λ_{\max} of the parent dye. The explanation of these shifts which fits in with the present theory is as follows. In the first place, as Brooker¹⁸ has shown, the magnitude of the bathochromic shift on ascending a vinylogous series decreases as the energetic asymmetry of the dye increases. The bathochromic effect of the introduction of an ethylidene group into indigo should, therefore, be greater than that for a similar structural change in the more highly asymmetric dye, thioindigo. The hypsochromic effect, which is clearly also brought into play by this change, is thus considered to be neutralised in the case of the indigo vinylogue but not in the case of the thioindigo vinylogue.

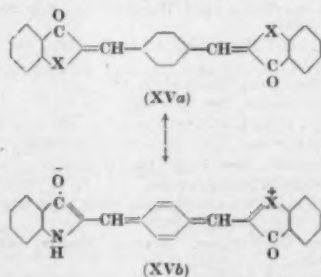
By introducing these carbon bridges into the dyes a number of further excited structures are also introduced. Such structures, e.g. XIIb, do not exist in the parent dyes, since the related one (e.g.

If), as has been discussed, may be neglected. They will be of considerably lower energy than these latter structures, and will consequently, according to the general rule¹, function hypsochromically. In normal dyes, such as cyanines, the lengthening of the conjugated chain introduces new excited structures the energies of which are comparable with those of existing ones (e.g. XIII and XIV, \bar{N} -replaced by $-\text{CH}-$), so that no hypsochromic effect is introduced. A complete analogy to this hypsochromic effect may be found, however, on proceeding from the bis-(1-ethyl-2-quinoline)methincyanine cation (XIII) to the related bis-(1-ethyl-2-quinoline)- α -azatrimethincyanine cation (XIV)¹⁹.



The excited structures shown are important¹ and may be compared with If and XIIb respectively. Because of the higher electronegativity of nitrogen compared with carbon, XIV will be more significant in its hybrid than will XIII in its hybrid. The effect of introducing the azomethine $=\text{CH}-\text{N}=\text{}$ link into XIII will therefore be to introduce a strong hypsochromic effect together with the bathochromic effect of the increase in the length of the chromophoric chain. The former effect predominates, however, and whereas λ_{max} of XIII is at 522 $m\mu$, that of XIV is at 519 $m\mu$. The bathochromic shift on proceeding from XIIa (X = S, n = 1) to XIIa (X = S, n = 2) now indicates that the effect of the increase in the length of conjugation predominates, since the energy levels of the newly introduced excited structures will be comparable with existing one (e.g. XIIb).

Friedländer and Risse¹⁷ also found that on interposing a phenylene group in XIIa (n = 1) λ_{max} shifts hypsochromically to 519 $m\mu$. when X = NH and to 488 $m\mu$. when X = S. These strong shifts of 80 $m\mu$. and 20 $m\mu$. respectively are not unexpected, since, although the length of conjugation is increased considerably, the energetic asymmetry of the molecules is greatly increased in that the transition from the lower-energy classical structure (XVa) involves loss of Kekulé resonance in the



phenylene bridge¹⁵. Moreover, a scale drawing of XVa (Fig. 1) shows a degree of overcrowding which may be relieved by rotating the

end nuclei around the predominantly single bonds attaching them to the phenylene ring. This will increase the single-bond character of these links, thus favouring structure XVa and separating still more the extreme levels¹⁵.

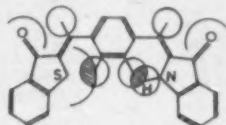


FIG. 1

EFFECT OF SUBSTITUENTS

Friedländer and Risse¹⁷ have shown that, whereas λ_{max} of indigo in xylene lies at 595 $m\mu$, that of *N*-methylindigo shifts bathochromically to 639 $m\mu$. and that of *NN'*-dimethylindigo to 644 $m\mu$. As Hodgson⁹ has pointed out, the latter may contain no hydrogen bonds and the former only one, and yet in spite of this both dyes are deeper in colour than the parent. The reason for this may be twofold. In the first place it is well known (see e.g. Förster⁸) that alkylation of amino auxochromes results in an increase in their $-M$ effect. This favouring of the higher-energy dipolar extreme structure will thus offset the increase in energetic asymmetry resulting from loss of hydrogen bonding. In the second place, as the scale drawing (Fig. 2) shows, alkylation causes considerable overcrowding of the *trans*-form (and also of the *cis*-form), so that loss of nuclear coplanarity by twisting of the bridge bonds must be assumed to occur. This will work bathochromically not only by decreasing the overlap of the C_2-C_2 π -orbitals but also by favouring the dipolar extreme structures, in which the bridge bond is

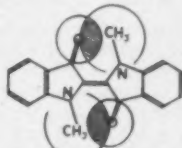


FIG. 2

single in character²⁰. Most of the overcrowding will occur on monoalkylation, there being more space for the accommodation of the second alkyl group in the distorted molecule. Consequently a larger shift on proceeding from indigo to the *N*-methyl derivative (44 $m\mu$.), than from the latter to the *NN'*-dimethyl derivative (5 $m\mu$.), would be expected, as is the case. Scheibe, Dörfling, and Assmann²¹ give ϵ_{max} 1.7×10^4 for indigo and 1.9×10^4 for *N*-ethylindigo. This may be taken as an indication that the decrease in ϵ_{max} by the reduction in the π -orbital overlap occasioned by twisting has been more than offset by the increase in ϵ_{max} due to a reduction in the non-degeneracy of the system²⁰.

Similar considerations also readily explain the relative instability and deeper colour of the non-

planar *NN'*-diacetylidindo compared with *NN'*-oxalylidindo.

Table I is drawn up from data given by Ettinger and Friedländer²² and by others.

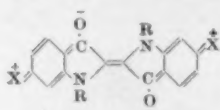
TABLE I

Dye	λ_{\max} (m μ)	Shift (m μ)	Dye	λ_{\max} (m μ)	Shift (m μ)
INDIGO	600	—	INDIGO	595	—
5:5'-Dibromo- ...	621	+21	5:5'-Dinitro- ...	585	-10
6:6'-Dibromo- ...	585	-15	6:6'-Dinitro- ...	635	+40
5:5'-Dimethoxy- ...	644	+44	<i>NN'</i> -DIMETHYL- INDIGO ...	644	—
6:6'-Dimethoxy- ...	577	-28	5:5'-Dichloro- ...	665	+21
			5:5'-Dimethyl- ...	665	+21
			6:6'-Dibromo- ...	638	-6

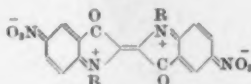
* In tetrachloroethane.

† In xylene.

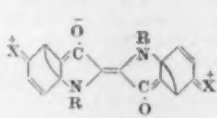
From this table it will be noted that the introduction of $-M$ substituents into the 5:5'-positions always results in a bathochromic shift, whilst similar substituents in the 6:6'-positions cause hypsochromic shifts. On the other hand, a $+M$ substituent in these positions gives shifts in the reverse directions. It can be shown that both types of substituents in any position will tend to deepen the colour of the dyes by functioning as auxiliary auxochromes, i.e. by rendering more significant those excited structures such as Id, e involved in the extraconjugation. However, particularly in their active positions, $\pm M$ substituents will function hypsochromically by increasing the energetic asymmetry of the molecule. By "active positions" are understood those positions in which the maximum $\pm M$ effect of the group may function. Thus a $-M$ substituent in the 6-position is active in that the resonance system represented by the classical structure and XVI will be more significant than the resonance system of which



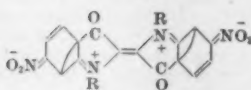
(XVI)



(XVII)



(XVIII)



(XIX)

XVIII is an extreme structure, in which the $-M$ group is in the 5-position. This follows from the fact that XVI has two covalent bonds more than XVIII. Similarly a $+M$ substituent is active in the 5-position (XVII) and not in the 6-position (XIX). It is obvious that the effect of increased contributions by structures such as XVI and XVII will be to cause a hypsochromic, increased separation of the energy levels of the extreme structures of the Arndt-type resonance, since the energy associated with these newly introduced systems must first be overcome in order that the dipolar structures (Ic) may contribute. Consequently it is found that the bathochromic effect given above is overcome only when the $\pm M$ atom or group is in its active position.

RESEARCH LABORATORIES

KODAK LTD.

WEALDSTONE, MIDDLESEX

(Received 13th December 1950)

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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Dyeing of Nylon

The Editor

Sir,

In the April issue of the *Journal* Dr. Douglas¹ states that "direct dyes, apart from a few which almost approach acid dyes in structure, yield low colour values or only tint nylon".

In the 1943 *Journal* I reported² some work done at Droylsden by Wilcock and myself which showed that the use of β -naphthol in an acetic acid bath

would give 100% exhaustion of some direct dyes, e.g. Chlorazol Blue B, whose structure I should assume is accepted as that of a typical direct dye.

This only followed what is obvious with all the new synthetic fibres, that they must first be swollen in order to take up some classes of dyes in reasonable quantities.

Yours truly

C. M. WHITTAKER

¹Douglas, *J.S.D.C.*, **67**, 133 (April 1951).

²Whittaker, *ibid.*, **59**, 69 (1943).

Notes

Proceedings of the Council

At a meeting of the Council, held at the offices of the Society, 32-34 Piccadilly, Bradford, on 13th June 1951, the proceedings included the following items of interest—

SOCIETY'S MEDALS COMMITTEE—The resignation of Mr. N. G. McCulloch was accepted with regret, and it was resolved that the thanks of Council for his past services be conveyed to him.

COLOUR INDEX (SECOND EDITION)—It was reported that there had been a very cordial and frank discussion of the problems involved in the joint production of the new *Colour Index* at a meeting held in Manchester on 29th May 1951 between the A.A.T.C.C. and the Society. The former had been represented by Messrs. C. Norris Rabold (President), W. D. Appel, and L. S. Little (Chairman of the Executive Committee on Research); the Society had been represented by the President, the Honorary Treasurer, the Honorary Secretary, and members of the Finance Committee. The opportunity for this exchange of views was very welcome, and it was gratifying that complete harmony had been reached on the wording of a draft agreement between the two bodies.

MEMBERSHIP—Six applications for ordinary membership and three for junior membership were approved.

Meetings of Council and Committees July

Council—No meeting

Finance—11th

Publications—17th

Colour Index Editorial Panel—2nd

Fastness Tests Co-ordinating—25th

Terms and Definitions—12th

Diploma—16th

"Tinctorial Arts To-day" Conference—26th

Mercer Lecture—30th

Second George Douglas Lecture

Arrangements have been made for the second George Douglas lecture to be given by Dr. R. Hill at the Midland Hotel, Manchester, on Friday, 7th March 1952. Dr. Hill will survey recent developments in the field of synthetic fibres.

Bournemouth I.S.O. Meeting on Fastness Testing

The second meeting of the International Standards Organisation Committee on Textiles was held in Bournemouth on 4-9th June 1951, when a wide range of subjects was discussed including colour fastness. The Society was represented by—

Mr. J. Barritt

Mr. J. Boulton

Dr. P. W. Cunliffe

Mr. J. G. Grundy

Mr. G. S. J. White

Mr. E. Wilson

At the first meeting, held in Buxton in 1948, it was agreed that a Joint Secretariat—the United Kingdom and the United States of America—should be responsible for work on colour fastness, the official organisations representing these countries being the British Standards Institution and the American Standards Association. In practice these bodies have delegated the technical work involved to the Society of Dyers and Colourists and the American Association of Textile Chemists and Colorists respectively.

At the Buxton meeting it was agreed that the fastness of dyed textiles to light, washing, and perspiration should first be examined. Official progress between 1948 and 1951 was slow, but the various organisations concerned with fastness testing on the Continent and in the U.K. and the U.S.A. were active in developing methods for assessing the fastness of dyed textiles to various agencies, and thus at the Bournemouth meeting a considerable amount of information, including published methods of fastness testing, was available.

The meetings on colour fastness were held on 7th and 8th June, and the following countries were represented—

Australia	Netherlands
Belgium	New Zealand
Canada	Norway
Czechoslovakia	South Africa
Denmark	Sweden
France	Switzerland
India	United Kingdom
Italy	United States of America

Four meetings of the Subcommittee ISO/TC 38/SC 1 Colour Fastness Testing were held, each of about three hours' duration and there was also a short plenary session of the Conference, at which the findings of the Subcommittee were reported. In practice, the Subcommittee comprised almost all the delegates whose primary interest was in colour fastness; it was noteworthy that from the beginning of the meetings the atmosphere was most cordial, and this was maintained throughout the sessions. Considerable progress was made, and at all times there was a desire for agreement.

A notable advance was the agreement to institute a Grey Scale for the measurement of colour loss or fading of dyed materials; this will comprise a series of five contrasts, increasing on a geometric visual scale. A small subcommittee was set up to discuss details of the work which will have to be carried out to implement this proposal. This subcommittee, comprising representatives from India, the Netherlands, France, the U.K., and the U.S.A., will proceed with the work immediately, maintaining close contact during its progress. When the Grey Scale is finally agreed upon, it is expected that progress in the consideration of many other fastness tests will be facilitated.

Considerable time was spent on the important question of light fastness, and it was agreed that there should be a standard daylight test using one set of eight blue standards which could also be used in fading lamps. The fastness organisations represented by the A.A.T.C.C., the Continental group, and the Society agreed to appoint representatives to form a small working party with the object of unifying the standards. Other countries agreed that they would accept the recommendations of this subcommittee. The U.S.A. delegation undertook to submit proposals for a standard lamp test.

In considering fastness to washing, it was agreed that the Grey Scale should be used for the evaluation of test results. As a step towards international agreement, conditions for mild and severe laundering were specified, and the Joint Secretariat agreed to circulate to other member countries the proposed methods of test together with an appropriate range of samples for such tests.

Progress on the unification of the perspiration test was made by the agreement to adopt the plate method, although considerable experimental work is required to determine the detailed conditions of test and the solutions to be used.

Agreement in principle was reached on the following tests, complete agreement in detail being achieved for most of them—

Acid spotting	Potting
Alkali spotting	Rubbing
Carbonising	Sea water
Hot pressing	Soda boiling
Meccerising	Stoving
Peroxide bleaching	Water spotting

It was also agreed that a test was required for fastness to organic solvents in general and that member countries would continue to work on this subject.

All the Society's delegates took an active part in the Conference, Dr. P. W. Cunliffe acting as spokesman and Mr. J. Barritt as Chairman of the subcommittees and the plenary session.

It is a pleasure to record the happy relations which existed between our partners the A.A.T.C.C., the members of the Continental fastness organisations, and our own members. The Society's delegates came away with the impression that real progress had been made both in matters of principle and in detailed agreement on many tests. Above all, they valued the opportunity of meeting delegates from other countries and discussing in a friendly atmosphere the various viewpoints on problems of colour fastness.

J. B.
P. W. C.

LONDON SECTION

Report for the Session 1950-51

The London Section is able to report with pleasure that activities have been well maintained during the past year. We have again enjoyed the privilege of holding our lectures in the rooms of the Royal Society; these meetings have been well attended, the average number present being 43. As is customary for this Section, with its great diversity of interests among members, the syllabus of lectures covered a very wide field, and, judging by the extent of the discussions which followed each lecture, we have been successful in both arousing and holding the interest of our members, whatever may be their particular branch of the colour-using industry. Details of the lectures are as follows—

6th October 1950	
<i>The Dyeing of Terylene and Ardl</i>	H. W. Speke
3rd November 1950	
<i>The Dyeing of Leather</i>	J. W. Stradwick
1st December 1950	
<i>The Physical Measurement of Dyeings, with special reference to the Ellis Photometer</i>	Dr. H. W. Ellis
5th January 1951	
<i>The Dyeing of Silk and Silk Unions</i>	A. Thomson
2nd February 1951	
<i>The Dyeing and Finishing of Rayon Warp Knitted Fabric</i>	J. Bromley and A. Cheek
2nd March 1951	
<i>A Survey of the Results of Recent Research in Dyeing and Finishing</i>	H. A. Turner
13th April 1951 (Luton)	
<i>Some Properties of Wool and their Industrial Significance</i>	Prof. J. B. Speakman

Apart from the normal six monthly lectures, this Section held a meeting at Luton in April, when the audience had the pleasure of hearing a lecture from Professor Speakman. The hat- and felt-making industries were well represented at this meeting, and although the room was a large one, every available seat was taken, there being over 100 people present.

As regards the social side of the Section's activities, these took the form of a Dinner-Dance at Oddenino's restaurant in February, to which members and their ladies were invited, and an informal dinner for members only, following our Annual General Meeting in April. Both these functions were well supported, and it is evident that the

opportunities so given to enable members to get to know each other better were greatly appreciated.

H. W. ELLIS

Honorary Secretary, London Section

Twenty-fourth International Congress of Industrial Chemistry

Paris, 25th November–1st December 1951

This congress will be held in conjunction with the first Salon de la Chimie (22nd November–2nd December), which will include equipment, plant, and products. The nineteen sections of the congress will include—

- 1—Analytical chemistry; laboratory equipment and apparatus
- 3—Water: treatment of drinking and industrial water; purification of effluents
- 11—Industrial organic products: dyes, etc.
- 12—Essences and perfumes; cosmetics; fats; soaps and detergents; etc.
- 13—Natural and artificial resins; rubber and synthetic elastomers; paints, pigments, and varnishes; inks
- 14—Cellulose, paper; natural and artificial textiles; bleaching, dyeing, printing, and finishing
- 15—Dyeing and tanning extracts; tanning industries; glues.

Full details and forms of application for admission can be obtained from the Société de Chimie Industrielle, 28 rue Saint-Dominique, Paris VII, France. Completed manuscripts of papers should reach the same address not later than 15th September 1951.

International Union of Leather Chemists' Societies

Second International Conference
London, 9–14th September 1951

This will be held at the Portland Hall Annexe of the Regent Street Polytechnic (entrance in Little Titchfield Street, off Great Portland Street, London W.1). Those intending to be present at the Conference should send their completed registration form to Mr. G. H. W. Humphreys, Forestal Central Laboratories, Harpenden Rise, Harpenden, Herts.

Patent Office Library

Extended Hours of Opening

The Patent Office Library, at 25 Southampton Buildings, Chancery Lane W.C.2, is now open to the public from 10 a.m. until 9 p.m., Mondays to Fridays inclusive, instead of closing at 6 p.m. as previously. Saturday opening, however, will continue to be from 10 a.m. to 5 p.m.

Technical Information and Documents Unit Transfer from B.O.T. to D.S.I.R.

The Technical Information and Documents Unit (T.I.D.U.), which for a number of years has been attached to the Board of Trade, has become part of the Information Services of the Department of Scientific and Industrial Research. For the present the Unit will remain in Lacon House, Theobalds Road, London W.C.1, where it will continue to hold at the disposal of industry a large collection of unpublished documents.

A small Technical Section is available to assist in the selection of material to meet specific needs, whilst a reading room is provided for those wishing to make a detailed study of drawings and documents. Alternatively, photocopies can be supplied at moderate charges.

Japanese Dye Production

The annual capacity of Japan's synthetic dye-making plant is about 18,000 tons, with a potential capacity of 19,660 tons. The figures for the different groups of dyes are—direct dyes 4000, acid dyes 800, basic dyes 600, chrome dyes 500, sulphide dyes 9000, Naphthol G 900, Naphthol D 900, and miscellaneous products 1200 tons.

A Seventeenth Century Pattern Book of Kendal "Cottons"

A pattern book containing 400 well preserved squares of cloths of various designs has been presented to the Corporation of Kendal, Westmorland, by Mr. J. O'Connor of that town. The book, the earliest decipherable date in which is 1678, was discovered in an attic in Kendal during the war-time hunt for salvage. The patterns in the book are Kendal "cottons". They include many examples of "ermine" or "spotted cottons", i.e. cloths spotted with red, blue, or green. C. O. C.

New Books and Publications

Veredler Jahrbuch

Deutscher Färbekalender 1951

Edited by O. Mecheels. Pp. lxxxii + 320. Munich: Franz Eder Verlag. Price, \$2.00.

Our readers cannot fail to be interested in the recovery of the German dye industry, and this book affords an easy method of keeping in touch with the expansion and development of both German and Swiss dyemaking firms.

Impressive lists are given of new products or of pre-war products once more obtainable, together with a brief description of their uses, etc. Interesting articles on various aspects of dyeing appear,

including a table of the effects of urea-formaldehyde on the hue and fastness to light of the Sirius Supra and Sirius ranges of dyes*. Readers interested in the azoic dyes will find constitutions given, but no more than are available in the BIOS and FIAT reports. Considerable space is given to fibres in admixture; methods for their identification are given, and the effects of the new fibres on garment dyeing are discussed.

This book, in German, is a handy and useful book of reference.

A. THOMSON

* These articles will be listed in the Abstracts section of a future issue of the *Journal*—Editor.

Organic Chemistry

By I. L. Finar. Pp. xv + 696. London: Longmans, Green & Co. 1951. Price, 40s. 0d.

The chemists of this country have for so long been dependent for text-books of organic chemistry upon translations of Continental publications that the recent policy of publishers in this country to encourage British authors is warmly welcomed. The present book differs from many post-war British books in this branch in endeavouring to provide a suitable text-book for those students aiming at General Honours degrees and Part I of the new Special Honours degree of London University. It should, therefore, by its scope, have provided a suitable basis for those engaged in a course of dyeing and its necessary organic chemistry.

This book certainly covers a large amount of ground, mainly in an orthodox, factual manner. The author claims to introduce and use electronic theories as early as possible, and the second chapter does give a brief account of the basic electronic theory of valency, and a non-mathematical introduction to molecular orbitals, without, however, any development on application of π -bonds. The constant application of these methods seems uneasily superimposed upon the more orthodox treatment generally used.

The book is full of detailed facts and covers a very wide range of the standard aliphatic, aromatic, and heterocyclic fields, but, except for its excellent treatment of tautomerism, very rarely gives sufficient descriptive matter to allow the reader to become attracted to and absorbed in the subject. Mention is made throughout of the latest facts and findings to introduce the reader to these topics, but the somewhat encyclopaedic method of presentation prevents the introduction from ever leading to an interest.

When, however, the reviewer turned to the topics of Dyes and Fibres, he found a complete failure on the part of the author to present even an accurate, let alone an up-to-date and adequate treatment. Terse, inaccurate descriptions of terms and processes follow an over-condensed treatment of the Relation between Colour and Constitution. Sulphur dyeing is disposed of in four lines, without mention of reduction, and to quote the whole detail on "rayon dyes"—"Rayons (p. 378) cannot be dyed in the usual way; a special technique is required".

Reference to p. 378 shows that the author is still of the "artificial silk" school, and that cellulose nitrate is the most important (judged by the space allotted) rayon of four described in less than one page.

The dyes selected for detailed treatment are the earliest basic and direct azo dyes, with no mention of azoics at all. Anthraquinone dyes are exemplified solely by Alizarin, and at the end there is printed the structural formula of Monastral Fast Blue BS, with the sole statement that this is the first commercial phthalocyanine dye.

One has the feeling that the antiquated and inaccurate statements in this most unsatisfactory chapter may not be the only ones in the book, and a doubt is thereby left in the mind of the reader as

to the veracity of other technical details throughout the book. It cannot be recommended for those engaged in dyeing or dyemaking. R. L. ELLIOTT

Technique of Organic Chemistry

Volume V

Adsorption and Chromatography

By H. G. Cassidy (edited by A. Weissberger).

Pp. xix + 360. New York and London: Interscience Publishers. 1951. Price, \$7.00 or 56s. 0d.

This latest addition to the well known series edited by Weissberger is the first to be written by a single author; the declared objective of the book is to describe and explain the various methods by which adsorption may be utilised in organic chemistry as a tool for the separation of mixtures. Chapter I (Definitions—Molecular Aspects of Adsorption) and Chapter II (Measurement of Adsorption. Treatment of Data) deal with the broader physicochemical aspects of the phenomena of adsorption in all the important binary systems (solid-gas, vapour-liquid, liquid-liquid, and solid-liquid). Considerable space is then devoted to an account of work relevant to an attempt at correlation of adsorbability and such factors as (a) solubility of adsorptive or polarity of solvent and adsorbent (Chapter III) and (b) the chemical and physical properties of the adsorptive (Chapter IV); this succeeds in so far as it is able, although, as the author points out, no general correlations can be expected. The correlations which can be made are usually limited to homologous series, but these can be of value when it is necessary to separate members of a series from one another, after having separated the class as a whole from other classes by alternative means. Chapter V (Summary of Relations) looks at the problem of separating mixtures from a different viewpoint, helps in the choice of solvent and adsorbent, and concludes with five useful "rules" predicting the relative extent of adsorption. Chapter VI (On the Nature of Separation Processes) gives a generalised survey of the mechanism of all separation processes.

The next four chapters are concerned with detailed aspects of Batchwise Adsorption and Decolorisation (Chapter VII), Chromatography (Chapter VIII), Ion-exchange Methods (Chapter IX), and Partition Chromatography (Chapter X). In addition to brief accounts of the well known aspects of adsorption chromatography and partition chromatography, these sections give much useful information on allied matters; the chapter on ion-exchange phenomena is devoted largely to a discussion of the mechanism of this process. Sections on "Applications" have evidently been kept deliberately small. The last chapter gives details for experiments which exemplify each of the chromatographic techniques, and concludes with a short section on the solution of problems of separation.

One could take exception to such words as "deashed" (for ashfree), "armamentarium", and "subsumes", but such occur very infrequently; few typographical errors were noted. All the credit for the invention of chromatography is given to Tswett (pp. 207-209), though recent publications have shown that earlier work by Day and others

foreshadowed Tswett's contribution. In the activation of alumina, a temperature of 200°C. can certainly be exceeded without loss of activity (p. 205). These criticisms become trivial, however, when considered in relation to the book as a whole; those concerned with problems involving separation of substances by adsorption phenomena will find that it helps to a better understanding of the physicochemical principles, and that it stimulates the use of such principles to better effect in practical applications to specific cases.

The book is well produced in conformity with the other volumes of the series; it is sufficiently well illustrated (though Fig. VIII-11 is unnecessary), contains numerous references, and is well indexed. Cumulative indexes are given on Authors and Subjects in Vol. I-V. F. R. CROPPER

A.S.T.M. Standards on Industrial Water

Prepared by A.S.T.M. Committee D-19 on Industrial Water. Pp. vii + 160. Philadelphia: American Society for Testing Materials. 1951. Price, paper, \$2.00.

This second edition of collected analytical and sampling procedures of the A.S.T.M. will be welcomed by chemists dealing with water treatment problems. Additions in this edition include methods for determination of aluminium, electrical conductivity, hardness, and sodium and potassium, together with a scheme for identification of types of micro-organisms in industrial waters. The method for identification of crystalline compounds in water deposits by X-ray diffraction has been completely rewritten, the revised instructions being very explicit, and there are new specifications for artificial sea water and definitions of terms encountered in water treatment.

The methods for chemical radicals are usually available elsewhere, but it is of value to have specific instructions for tests, some of which are not regularly carried out in all laboratories. It is, however, in the field of special tests such as those for corrosivity and micro-organisms that the volume will be of most service. The new specification on identification of micro-organism types follows the high standard set by previous standards, and a method for identification of algae is being prepared.

The hardness determination is by the soap method and includes a "lather factor" procedure. It is as yet too early to expect an official pronouncement on the newer "complexone" methods, but in view of the slightly varying techniques developed in different laboratories, the early attention of the committee would be particularly valuable.

The selection of terms requiring definition is not without its difficulties and, although there are obvious possibilities for extension, the present list is well chosen. It is, however, debatable whether the definitions should not preferably have been incorporated in the respective specifications on reporting analytical results.

The interval between the two editions is only fifteen months and, as a number of important tests remain for attention, frequent revision may be anticipated. Most chemists will probably not wish,

therefore, to subscribe to every edition, although the cost of two dollars for the present volume is not unduly high.

C. W. DRANE

Monomers

A Collection of Data and Procedures on the Basic Materials for the Synthesis of Fibres, Plastics, and Rubbers (Section II)

Edited by E. R. Blout and H. Mark. New York and London: Interscience Publishers. 1951. Price, \$5.00.

- (i) *Acrylic Acid* (J. T. Clarke; pp. 19; 61 references).
- (ii) *Esters of Acrylic Acid* (J. T. Clarke; pp. 37; 97 references).
- (iii) *Esters of Methacrylic Acid other than Methyl Methacrylate* (R. S. Corley; pp. 86; 128 references).
- (iv) *N-Vinylcarbazole* (K. C. Frisch; pp. 25; 46 references).
- (v) *Vinyl Ethers* (C. E. Schildknecht; pp. 59; 159 references).
- (vi) *Vinylpyridine* (H. Frisch; pp. 31; 48 references).

These six photolithographed booklets for filing in the original binder bring to fourteen the chapters on single monomers or groups of monomers of importance for the making of high-molecular polymerisation and polycondensation products in the series initiated by the publishers in 1949, and are issued with an Errata sheet of corrections to be made in the first eight chapters. In scope, the new chapters are similar to the earlier ones, dealing with methods of preparation suitable for laboratory use and for industrial production, toxic and other hazards, analysis, physical properties, chemical reactions, and polymerisation.

In a very general way, these booklets provide the kind of literature reviews that a chemist might make for himself when embarking upon work in these fields, but different degrees of experience clearly lie behind their composition. To those expert in the various fields, the value of the booklets will undoubtedly reside mainly in the collections of physical data, which seem to be fairly reliably quoted. Those not so expert will find much more of interest, but they will feel that the intentions of the editors have not always been realised: thus, instead of "the one or two best methods of preparing small quantities", they will often find an array of methods; looking for information on impurities, purification, analysis, handling, they will often be disappointed.

There is in these booklets no homogeneous bibliographical treatment. References are sometimes arranged alphabetically, sometimes according to the compilers' development and afterthoughts; patentees' names are not always given; all authors do not give Chemical Abstracts references to patent specifications; "ibid." is used half-heartedly, and not always correctly; authors' names are not always correctly quoted (Moureu is several times rendered as Moreau); but, in a selection tested, the reviewer was invariably directed to the proper page of a journal, or to the correct patent specification, after

having on occasion corrected the reference number cited in the text.

There are many other errors and misprints to be corrected in future editions; blunders in chemical formulae will be noted on pp. 17, 26, 29, 43, 50 of (iii).

Careful editing and more critical treatment of the subject-matter would have enhanced the appeal of these publications considerably; but, despite their many blemishes, they are undeniably useful to anyone with a practical interest in polymers.

W. CHARLTON

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes.
Any publication abstracted may be referred to by members of the Society on application to
Dr. C. B. Stevens, Dyeing Department, Leeds University

Allied Colloids (Bradford) Ltd.

TEXTILE AUXILIARY PRODUCTS—The following circulars have been issued by the Badische Anilin- und Soda-Fabrik (B.A.S.F.), Ludwigshafen. They deal with either the properties and general application of an individual product or its use for a particular purpose—

ANTHRAQUINONE POWDER, PASTE 30%—Reduction accelerators in discharge printing.

BASOPAL NA CONC. POWDER—An aryalkylsulphonate developed as a synthetic detergent.

DISSOLVING SALT B—A salt of an aromatic sulphonic acid designed for use in textile printing to facilitate penetration and increase colour yield.

DISSOLVING SALT CG—A mixture of solvents primarily recommended for increasing the rate of fixation of chrome dyes in textile printing.

GLYCINE A—An auxiliary product for textile printing.

INDANTHREN BLUE GPZ POWDER FINE; RPZ POWDER FINE—Dyes for blueing all types of textile materials. The blueing effect obtained is exceptionally fast to light and completely unaffected by acids, alkalis, or salts.

LAVENTINE KB—A mixture of wetting agents, dispersing agents, and solvents for degreasing and cleaning textile materials.

LUMATTIN SL (formerly Dullit SL)—This is a substantive delustering agent for regenerated cellulose rayon consisting of a white pigment in combination with a cation-active dispersing agent.

NEKANIL W EXTRA—A non-ionic detergent for wool processing.

PALATINE FAST SALT O SOLUTION—An auxiliary product for adding to the dyebath when applying pre-metallised dyes to make possible a reduction in the amount of sulphuric acid required.

PEREGAL OK SPECIAL—A cation-active levelling agent for vat dyes.

PEREGAL ON—A non-ionic auxiliary product recommended as a levelling agent for all dyes, but particularly for vat dyes.

RONGALITE CL—A mixture of Rongalite C, Leucotrope W, and sodium carbonate for discharge printing, particularly on vat-dyed grounds.

SERODIT D—An organic sulphonic acid which may be used instead of mineral acid for producing local carbonisation. It is very conveniently applied by means of roller, screen, or block, and any cellulosic material is destroyed by steaming for 10–15 min. The degradation products are readily washed out. Vat dyes may be printed alongside Serodit D pastes, and certain dispersed and premetallised dyes incorporated in the paste itself to give coloured effects on the non-cellulosic portion of the fabric.

SIZING AGENT TS—A copolymer for sizing materials composed of acetate rayon, nylon, and polyvinyl chloride.

TEXTAPEET NA (formerly Appretan NA)—A water-soluble synthetic resin for finishing all types of woven fabrics.

THICKENING HD—A water-soluble cellulose ether used as a thickening agent in textile printing.

INDANTHREN DARK BLUE DB POWDER—This anthraquinonoid vat dye, manufactured by the B.A.S.F., is recommended for use alone and in mixtures for producing navy blues of exceptional fastness to light on cellulosic materials. Dyeing method IN is recommended. Some fastness figures on cotton include—Light 8, soda boiling 4, chlorine 2–3.

INDANTHREN GREY CL POWDER—This anthraquinone vat dye is suitable for producing bluish greys on all types of cellulosic material. The IW dyeing method is recommended, and it may also be applied by the pigment padding and leuco acid processes. Mixtures of native and regenerated cellulose are dyed to practically the same shade. Some fastness properties on cotton include—Light 7, chlorine 4–5, soda boiling 4.

COLOURS FOR RUBBER—This pattern card, issued by the Société Anonyme de Matières Colorantes et Produits Chimiques Francolor, illustrates twenty-seven Vulcafix Colours for colouring rubber in the mass. Each is shown in two depths, and six mixture colourings using colouring matters having the maximum fastness to sunlight and suitable for all types of cures are also shown.

Reports on German Industry

I.G. Farbenindustrie A.-G., Ludwigshafen

FD 281–288, 290/51* (each of these is a part of microfilm PB 70,332).

CHROME, PALATINE, AND CELLULOSE NITRATE DYES FD 281/51*.

Microfilm copies of the following papers (author and date are given in parentheses)—

- (1) *Chrome Complex Dyes from Hydroxyquinolines* (Krzikalla, 23rd March 1935)
- (2) *Analysis of the Cellulose Nitrate Dyes of Ciba* (Kirsch, 12th June 1935)
- (3) *Chromable Triarylmethane Dyes derived from Alkylhydroxycyclic Acids—I* (Krzikalla, 15th April 1936)
- (4) *Chromable Diphenylamines and Acridones—I* (Krzikalla, 15th April 1936)

(5) *Dyeing with Palatine Fast Dyes in Weakly Acid Baths* (Krzikalla, 6th Nov. 1936)

(6) *Diffusion Measurements of Dyes. Particle Size and Dyeing Behaviour of Palatine Fast Dyes* (E. Valko, 21st Jan. 1937)

(7) *Chromable Diphenylamines and Acridones—II* (Krzikalla, 12th May 1937)

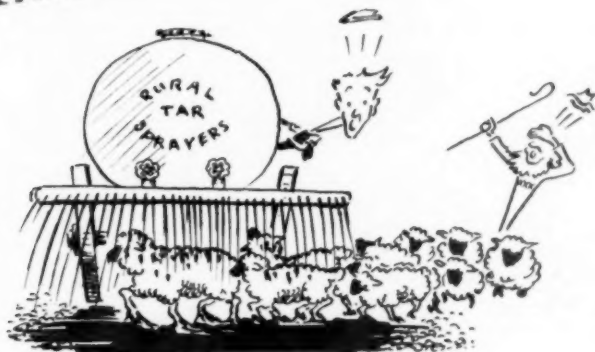
(8) *Chromable Blue Triphenylmethane Dyes from p-Formyl-o-cresotic Acid and Diphenylamine Derivatives* (Krzikalla, 12th May 1937)

(9) *Chromable Triphenylmethane Dyes from 1-Hydroxybenzene-2,6-dicarboxylic Acid* (Krzikalla, 13th May 1937)

(10) *Azomethines containing Chromium, a New Type of Palatine Fast Dyes* (Krzikalla, 15th May 1937)

* Material available for inspection at, and photocopies available from, T.I.D.U., D.S.I.R., Lagoon House, Theobalds Road, London W.C.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.

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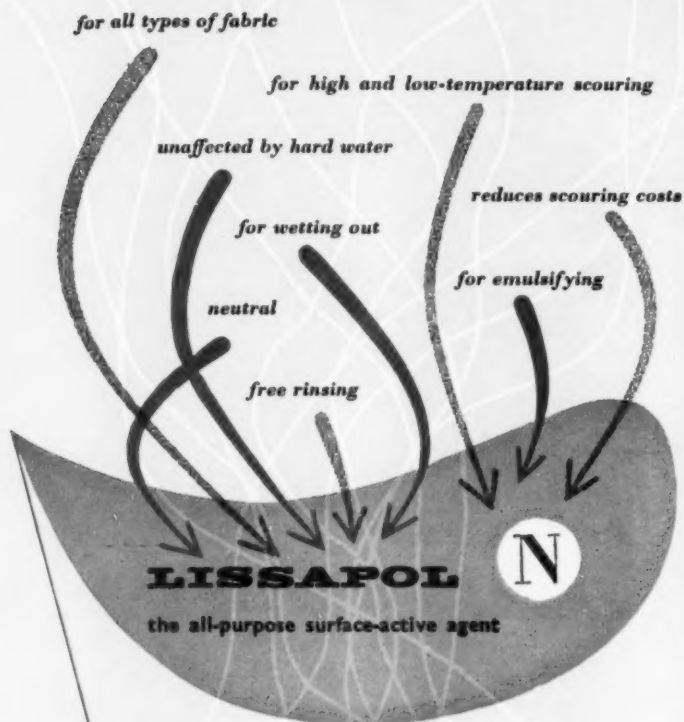
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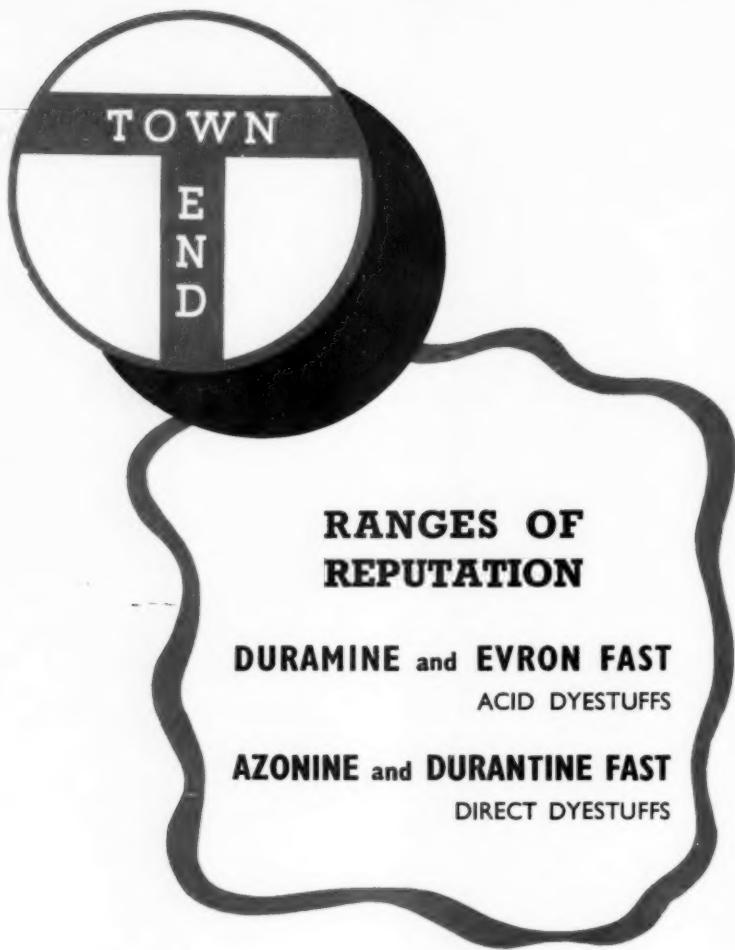
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- (11) *Chromable Triarylmethane Dyes from Aldehydo-salicylic Acids—II* (Krzikalla, 18th May 1937)—Chromoxane Brilliant Violet 2R is derived from *p*-aldehydo-*o*-cresotic acid and 2 mol. of *o*-tolyltaurine
- (12) *Chrome Complex Dyes* (Krzikalla, 21st June 1937)—Survey of knowledge at that date.

NAPHTOL AS AND AZABENZANTHRONE DYES

FD 282/51*.

- (1) *Oil-soluble Dyes containing Aliphatic Radicals* (Krzikalla, 26th March 1935)
- (2) *Vat Green P 401 and Other Work on the 11-Azabenzanthrone Series* (Köberle, 21st Feb. 1936)
- (3) *β -Azabenzanthrones* (Köberle, 24th Feb. 1936)
- (4) *Further Work on the 1-Azabenzanthrone Series* (Ebel, 3rd March 1936)
- (5) *δ -Azabenzanthrone* (Köberle, 17th Sept. 1936)
- (6) *Naphthols containing Sulphonyl Groups suitable for dyeing Wolletra* (Krzikalla, 6th Nov. 1936)
- (7) *Neutral Water-soluble Substantive Naphthols for Fast Developing Colours on Wolletra* (Krzikalla, 14th May 1937)
- (8) *δ -Azabenzanthrone* (Steigerwald, 18th Sept. 1937).

AZO DYES

FD 283/51*.

- (1) *2:3-Hydrozynaphthoylmethyltaurine as Coupling Component for Azo Acid Dyes* (Krzikalla, 26th March 1935)
- (2) *Azo Dyes from Hydroxytriphenylencarboxyaryldes* (Schuster and Schnabel, 27th March 1935)
- (3) *Azo Dyes for Two-tone Colours* (Krzikalla, 13th Nov. 1935)—Celliton Discharge Blue 3R was to be offered for this purpose as Sudan Blue R.
- (4) *Azo Dyes from Monophthalimide* (Krzikalla, 18th Oct. 1935)
- (5) *New Water-soluble Azo Dyes of Basic Character* (Krzikalla, 28th Nov. 1935)
- (6) *Dihydroresorcinol as Coupling Component for Azo Dyes* (Krzikalla, 4th Dec. 1935)
- (7) *Erganol Dyes based on Resorcinol* (Krzikalla, 3rd Nov. 1936)—Erganol Dark Brown CN was—
Picramic acid—Resorcinol—2-Toluidine-5-sulphonic acid
Erganol Medium Brown was—
Picramic acid—Resorcinol—2-Anisidine-4-sulphonic acid
- (8) *Nigger Brown Ferriferous Azo Dyes for Leather* (Krzikalla, 3rd Nov. 1936)
- (9) *Azo Dyes from *p*-Substituted Alkylphenols* (Krzikalla, 3rd Nov. 1936)—Acid Anthracene Brown LE was—
4-Nitro-*o*-aminophenol-6-sulphonic acid—
p-tert.-Butylphenol
its chrome complex was Palatine Fast Brown 21635.
- (10) *Azo Dyes from Aminophthalic Acid* (Krzikalla, 9th Nov. 1936)
- (11) *Water-soluble Acetate Rayon Dyes* (Krzikalla, 9th Nov. 1936)
- (12) *Sugar Compounds as Components for Acetate Rayon Dyes* (Schuster, 9th Nov. 1936)
- (13) *Azo Dyes from cycloHexylmethylpyrazolone* (Krzikalla, 10th Nov. 1936)
- (14) *Catalytic Reduction of Polynuclear Aromatic Compounds* (Eichel, 31st Dec. 1936)
- (15) *Dyeing Mixed Fibres with Substantive Diazo Compounds (Azo Compounds as Dye Intermediates)* (Ebel, 22nd Feb. 1937)
- (16) *Water-soluble Acetate Rayon Dyes* (Krzikalla, 24th May 1937)
- (17) *Azo Dyes from γ -Hydroxy- α -ketofulolin* (Krzikalla, 24th May 1937)
- (18) *An Igenal Dark Green of the C Series* (Krzikalla, 24th May 1937)
- (19) *Metal Complexes of Azo Dyes* (Eistert, 25th May 1937)

DYES FOR LEATHER, RAYON, WOOL, AND MIXED FIBRES

FD 284/51*.

- (1) *Pyron Dyes* (Krzikalla, 9th April 1935)
- (2) *Acridone Wool Dyes* (Krzikalla, 11th May 1935)
- (3) *5-Amino-1:9-anthrapyrimidine-2-sulphonic Acid as Starting Material for New Dyes* (Schlichting, 18th Oct. 1935)
- (4) *Dyes from 2:6-Diamino-1-chlorobenzene-4-sulphonic Acid* (Krzikalla, 12th Nov. 1935)
- (5) *Nitrodiphenylamine Leather Dyes of Good Penetration* (Krzikalla, 15th April 1936)
- (6) *Erganol Dyes from Benzidine-*o*-dicarboxylic Acid* (Krzikalla, 2nd Nov. 1936)
- (7) *Surface-dyeing Leather Dyes from 4-Nitro-1:3-phenylenediamine* (Krzikalla, 3rd Nov. 1936)—Igenal Yellow Brown J 836 was a mixture of Direct Yellow R extra and 5-nitro-2-anisidine-4-sulphonic acid—4-nitro-1:3-phenylenediamine.
- (8) *Leather Dyes of Good Penetration* (Krzikalla, 4th Nov. 1936)—Igenal Brown P2G was a mixture of acid dyes and the condensation product of 2:4-dinitrochlorobenzene-sulphonic acid and dimethyl-*p*-phenylenediaminethiosulphonic acid.
- (9) *Acetate Rayon Dyes (γ -Ketobutylaminoanthraquinones)* (Eichel, 31st Dec. 1936)
- (10) *Pressure Extract obtained from Coal [Sulphonated Derivatives as Leather Dyes]* (Schuster, 16th April 1937)
- (11) *Nitro-oxazines and -thiazines as Leather Dyes of Good Penetration* (Krzikalla, 13th May 1937)
- (12) *Erganol Dyes* (Krzikalla, 24th May 1937)
- (13) *Acid Wool Dyes having External Basic Groups* (Krzikalla, 28th May 1937).

ANALYSES OF COMPETITORS' DYES

FD 285/51*.

Thirteen reports on the analysis of many competitive products.

ANTHRAQUINONE DYES, INDIGOSOLS, LEUCO, AND PYRIMIDINO COMPOUNDS

FD 286/51*.

- (1) *Indigosols from 4-Aminoanthraquinone-2:1(N)-1':2'(N)-benzocridone Dyes* (Köberle and Münster, 15th Oct. 1935)
- (2) *Stable Leuco Compounds of the Leucamine and Leuco Quinizarin Type* (Steigerwald, 16th Oct. 1935)
- (3) *Preparation of Secondary, Mixed-substituted 1:4-Diaminoanthraquinones* (Köberle and Steigerwald, 16th Oct. 1935)
- (4) *Constitution of the Aminoanthrapyrimidin-sulphonic Acid obtained from 1:5-Diaminoanthraquinone-2-sulphonic Acid with Formamide* (Köberle, 17th Oct. 1935)
- (5) *Indigosols from Anthraquinoneazul Dyes [Indigosol Red IFBB]* (Köberle and Münster, 17th Oct. 1935)
- (6) *Indigosols from Pyrazoleanthranone Dyes* (Köberle, 17th Oct. 1935)
- (7) *1-Amino- and 1-Alkylamino-4-hydroxy-anthraquinones* (Steigerwald, 25th Feb. 1936)
- (8) *Research in the Naphthindione Series* (Köberle, 15th Sept. 1936)
- (9) *Phenylazoloanthraquinone Dyes* (Schlichting, 22nd Sept. 1936)
- (10) *New Method of producing Pyrimidino Compounds* (Köberle, 12th Oct. 1936)
- (11) *Work in the Indigosol Field* (Berti, 23rd Feb. 1937)
- (12) *Phenylazoloanthraquinone Dyes* (Schlichting, 25th Feb. 1937).

PHTHALOCYANINES AND PIGMENT, METALLIZABLE, FLUORESCENT, AND VAT DYES

FD 287/51*.

- (1) *Pigment Dyes on a Phthalodinitrile Basis* (Hopff and Steinbrunn, 8th Jan. 1936)
- (2) *Oil-soluble and Fluorescent Dyes* (Köberle, 4th March 1936)
- (3) *2-Benzoperylene* (Köberle, 9th March 1936)

* Material available for inspection at, and photocopies available from, T.I.D.U., D.S.I.R., Lacom House, Theobalds Road, London W.C.1, or (under FB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.

- (4) *Dyes of the 1:9-Anthracyridone Series* (Steigerwald, 21st Sept. 1936)
- (5) *Studies on Fluorescent Compounds* (Hopff, 7th Jan. 1937)—Fluorol 5GM was 3:13-dimethylcrotonene.
- (6) *Reaction of Halogeno Compounds with Cuprous Cyanide* (Köberle, 18th Feb. 1937)
- (7) *Perylenecarboxylic Esters* (Fluorescent Materials) (Schlichting, 22nd Feb. 1937)
- (8) *Pressure Chlorination of Phthalocyanines* (Wiest, 25th Feb. 1937)
- (9) *Pressureless Chlorination of Heliogen Blue B and G* (Diebold, 10th March 1937)
- (10) *Halogenophthalocyanines by the Sulphuryl Chloride Process* (Hopff, 16th April 1937)
- (11) *Importance of Five- and Six-membered Secondary Valency Ring Formation for the Stability of Metal-Dye Complexes* (Krzikalla, 20th May 1937)
- (12) *Molecular Mechanism of Pigment Washing* (Valko, 10th June 1937)
- (13) *Titanium Dioxide as White Body Colour* (Wurzschnitt, 8th July 1937)
- (14) *Linkage of Iron in Vulcan Green B extra* (Wurzschnitt, 1st Nov. 1937).

DYE INTERMEDIATES, DYE TESTS, AND CLEAFENED PROCESSES

FD 288/51*.

- (1) *New Auxiliary Agents for Dyeing [and Manufacture of Indanthren Blue RS powder fine]* (Ebel, 5th March 1936)
- (2) *Dependence of Hue of Heliogen Blue Coatings on Particle Size* (von Susich, 12th Aug. 1936)

- (3) *Aza Compounds as Dye Intermediates* (Köberle, 23rd Sept. 1936)
- (4) *8-Aminoquinoline* (Arnim, 26th Feb. 1936).

TEXTILE AUXILIARIES

FD 290/51*.

- (1) *Dodecyl Polyethylene Glycol Ether as Example in a Comparative Investigation into Non-ionic Textile Auxiliaries* (Günther and Valko, 30th Sept. 1935)
- (2) *Machine for Testing Washing Agents* (Hengstenberg, 25th Sept. 1936)
- (3) *Mechanism of Levelling Agents* (Valko, 8th Oct. 1936)
- (4) *Textile Auxiliaries from Chloroalkyl Polyglycol Ethers* (Götz, 12th Oct. 1936)
- (5) *Alcohols from Aryl Ketones and their use in preparing Textile Auxiliaries* (Schuster, 13th Oct. 1936)
- (6) *Non-slip Agent for Rayon* (Krzikalla, 13th Oct. 1936)
- (7) *Improvement in Ramasil K conc.* (Hengstenberg, 18th Dec. 1936)
- (8) *Crotonaldehyde Condensates* (Schmidt, 14th April 1937)
- (9) *Constitution of Water-soluble Calcium Compounds of Trilon A and Ma. 416* (Pätzner, 8th July 1937)
- (10) *Copolymers of Crotonic Esters* (Hopff, 12th Oct. 1937)
- (11) *New Condensates from Vinyl Methyl Ketone and Formaldehyde* (Hopff, 13th Oct. 1937).

C. O. C.

* Material available for inspection at, and photocopies available from, T.I.D.U., D.S.I.R., Lacon House, Theobalds Road, London W.C.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.

Abstracts from British and Foreign Journals and Patents

(The Titles of Patents are abridged and modified)

I—PLANT; MACHINERY; BUILDINGS

Control of J Boxes and Steamers in Continuous Bleaching.

C. W. Bowden Jr. and D. B. Daubert. *Amer. Dyestuff Rep.*, 40, P286-P292 (30th April 1951). Problems of control are discussed in relation to du Pont J boxes, Becco J boxes, and Mathieson steamers, the first and last being noted as relatively simple to control. The Becco unit requires closer attention to details of cloth level and external influences. Thermocouple location, mounting methods, and circuit wiring for direct, averaging, or differential temperature readings are described.

J. W. B.

A Flemish Dyehouse.

C. O. Clark. *J. Bradford Textile Soc.*, 34-35 (1949-1950). Comments, accompanied by a coloured illustration, on a picture, owned by the British Colour Council, of a church converted into a dyehouse. The picture was painted about 1810.

C. O. C.

PATENTS

Thread-advancing Reel.

American Viscose Corpn. B.P. 652,465, B.P. 652,719. A unitary reel of simplified construction that is economical to make and to maintain is described.

C. O. C.

Thread-advancing Reels.

Courtaulds Ltd. and T. S. Mayner. B.P. 652,973, B.P. 652,974. A reel is described on which more than one processing operation can be done while mingling of processing liquors is kept at a minimum.

Courtaulds Ltd. and K. M. McLellan. B.P. 652,975. A multistage reel by means of which the thread can be passed from one stage to the next with minimum distortion.

Courtaulds Ltd. and L. A. Corey. B.P. 652,976. A multistage reel on which the thread can be shrunk or stretched over part or the whole of the periphery of the

reel, if desired after one or more wet treatments, with minimum distortion.

Courtaulds Ltd. and A. W. Eldman. B.P. 652,977.

A multistage reel which permits the thread to travel over it with uniform pitch of the helix and with minimum distortion of the thread as it passes from one section of the reel to the next.

C. O. C.

Thread-advancing Reels.

British Celanese Ltd. B.P. 653,269.

Heat Setting.

Fair Lawn Finishing Co. B.P. 652,545.

Apparatus is described in which the material is passed through a zone where it is treated for 1-10 sec. by a high-velocity heating gas current whose temperature is predetermined by the material being treated; e.g. nylon fabric is treated in open width by a 400-4000 ft./min. current of gas at 400-475°F.

C. O. C.

Dye Jig.

A. Corrodi. B.P. 652,447.

Driving the rollers of a dye jig by direct contact with the counter-rotating primary and secondary members of an electric motor ensures constant tension on the fabric.

C. O. C.

Feeding Fabrics in Open Width.

Fair Lawn Finishing Co. and W. G. Helms. U.S.P. 2,533,247.

The feed slot of the below-bath-surface current-conveying type contains a convexly arcuate surface with a downwardly extending end-portion, this latter being placed in vertical directional alignment with the inlet end of the slot. Treating liquid passes continuously on to and over this surface toward and over the end portion. The fabric is fed on to this surface in open width and in absence of tension. This feeds the fabric into the bath without distortion or formation of creases and without entrapment of air bubbles.

U.S.P. 2,533,248.

The fabric is subjected to unrestricted shrinkage with a suitable liquor while in uniformly supported tension contact with a smooth surface; it is then immediately

relaxed in liquid and while relaxed conveyed by a stream of liquor over a downwardly arcuating surface into a vertical position, where it can be gripped by a below-bath-surface conveying current. C. O. C.

Application of Dyes and Other Materials by Spraying.

American Viscose Corp. and F. C. Wedler.

U.S.P. 2,532,471.

A primary heating chamber and a drying chamber are separated by one or more intermediate chambers. Each of the last-named has entry and exit passages in opposite walls and contains rollers, there being one roller close to each of the passages so as to guide fabric entering or leaving the chamber and in addition at least one other roller mounted on an axis displaced from the plane of the axes of the rollers close to the passages, so that the fabric is guided in a devious path through the chamber. Treating liquor is sprayed on to the fabric in the primary and intermediate chambers. C. O. C.

Stenters. W. W. Spooner.

B.P. 652,773.

In a clip or pin stenter one or both of the chain rails is automatically displaced in accordance with variations in width of the web as it becomes engaged by the clips or pins. By using a slow-moving mechanism of low inertia for displacing the chain rails there is no fear of their over-running as they reach their required new position. C. O. C.

Coating Paper. Champion Paper & Fibre Co. and W. P. Taylor.

U.S.P. 2,534,320.

Coating of controlled thickness is obtained on paper supported on a roll or belt carrier by use of a specially shaped and placed doctor blade and a carrier of hardness and resilience within specific limits. The blade has to press on the paper so that it deforms the surface of the carrier and causes the paper to adhere to it temporarily. U.S.P. 2,534,321.

The carrier surface is dampened before it comes into contact with the paper. This, together with the pressure of the doctor, gives enough adhesion to make the paper strongly resist any forces tending to cause it to slide along the carrier but not enough to prevent its readily parting from the carrier when pulled along a path which diverges therefrom. This permits of threading under doctor blades without lifting the blade or wasting coating held in front of the blade, and carrying through of buckles and slack edges, and prevents wrinkling and tearing. C. O. C.

Coating Paper. Minnesota & Ontario Paper Co., B. K. Asdell, and W. H. Barrett.

U.S.P. 2,534,848.

A machine which applies two coats to the same side of a rapidly moving web of paper. C. O. C.

Drying Paper, Fabrics, etc. Wall Paper Manufacturers Ltd., G. Keeble, Hunt & Moscrop Ltd., and J. Sharpe.

B.P. 652,044.

In a drying machine of the type described in B.P. 570,774 (cf. J.S.D.C., 62, 36 (1946)) the guide roller around which the material being dried travels when passing from the upper to the lower chamber is mounted on supports which yield when the material shrinks, thus preventing undue stresses in, and consequent breakages of, the material. C. O. C.

Drying Cylinders and Drying Stacks of Papermaking Machines. Millspaugh Ltd. and R. C. Heys.

B.P. 652,747.

A rotatable cylinder for the drier stack of a papermaking machine has a perforated shell containing internal boxes for applying suction and high-velocity air across the whole perforated width of the shell, the suction box extending over half the periphery and the air being applied beyond both ends of the suction box to allow it to be directed on the paper web as it meets and leaves the shell. A blank sector may exist between the two high-velocity air boxes or between the suction and air sectors. The suction box may have two or more longitudinal compartments to enable suction to be applied in stages or to greater or less portions of the periphery. One or more shoes occupying only part of the length of the cylinder, covered by the usual felt and adjustable across the width of the web, may be located opposite the periphery under which the suction box operates to allow hot air or radiant heat to be applied to dry wet streaks in the web, or may be arranged to apply hot air or radiant heat to part of the web as it passes from cylinder to cylinder. A presser roll may work in conjunction with the cylinder to express air, steam, or even condensed

water from the heated web of paper. A two-tier drier stack may include a perforated cylinder in each row with a short endless felt or wire for each perforated cylinder to prevent marking of the underside by the perforations in the cylinder. S. V. S.

Adjustable Fur Pelt Drier and Stretcher. T. A. Miskowski and E. Schulz, Sr.

U.S.P. 2,534,071.

Paper Pulp and Pulp-making Materials. C. W. Morden.

B.P. 652,439.

A machine for disintegrating and treating paper pulp, etc., comprises a tank in which are mounted a rotating disc and a stationary concentric ring. The disc has an annular rim the inner periphery of which is of the same diameter as the inner periphery of the ring. The opposed faces of rim and ring comprise attritioning surfaces, each having a set of shredding teeth extending inwardly towards the centre. One set of teeth is set obliquely and is thus not parallel with the vertical plane of the other set. The wall of the tank has a circular opening to which the inner marginal portion of the ring is attached, whilst the rotating disc is mounted in the opening adjacent to the outer end-face of the ring on a driving shaft. The annular passageway between ring and wall serves to convey the treated pulp back to the tank or through a valve-controlled conduit from the tank with the aid of a flow-directing ring. A flexible branch pipe enables the treated pulp stream to be directed to any part of the tank. S. V. S.

Automatic Control of the Sequence and Duration of Operations in a Laundry Washing Machine.

Pioneer Laundry Ltd. and W. D. McComb.

B.P. 653,084.

Fur-conditioning Machine. Reliable Machine Works Inc.

B.P. 652,291.

A machine is described which allows finished fur garments which are considerably larger than the conditioning drum to be efficiently treated. C. O. C.

Striping Machine for Wire. Surprenant Mfg. Co.

B.P. 652,769.

A machine for carrying out simultaneously several striping operations. C. O. C.

Carbon Black. Columbian Carbon Co. (IV, p. 319.)

II—WATER AND EFFLUENTS

Treatment of Sewage containing Textile Trade Effluents. W. H. Hillier. *J. Bradford Textile Soc.*, 96-98 (1949-50).

A brief account of the Bradford Corporation's sewage works at Esholt, reference being made to the difficulties caused by the presence in the sewage of synthetic detergents. C. O. C.

Characteristics and Treatment of Wool-dyeing Wastes. J. A. McCarthy. *Sewage & Industrial Wastes (Sewage Works J.)*, 22, 77-86 (1950); *Water Pollution Ab.*, 23, 211 (Sept. 1950).

Waste waters from wool dyeing are weaker than those from wool scouring, but 3-4 times stronger than domestic sewage. The colour, pH, alkalinity, biological oxygen demand (B.O.D.), and contents of dissolved and suspended solids, nitrogen, and fats of typical dye wastes are tabulated. Wastes from acid dyeing contain very little suspended matter, so neither coagulation nor sedimentation is effective. The waste waters have a suitable carbon : nitrogen ratio and contain considerable numbers of bacteria; results of treatment by percolating filters are given. Good nitrification and considerable reduction in B.O.D., grease, and colour were obtained with loadings of 1500-2000 lb. of B.O.D. per acre-ft. when pH was > 5.5. Below pH 5.5 maximum possible load decreased rapidly. Heavier loadings were possible when the effluent was recirculated. Vat dyeing effluents are weaker than those from acid dyeing and have pH approx. 9, which interferes with biological filtration. When acid and vat dye waste waters were mixed in approx. equal proportions, satisfactory results were obtained with percolating filters operated at loads up to 1500 lb. of B.O.D. per acre-ft. The waste waters could be treated successfully when mixed with domestic sewage. Only a small volume of sludge is produced by treatment of these waste waters; it dries rapidly and is easily digested. C. O. C.

Treatment of Cotton Waste Waters in India. S. Balasundaram and V. Subrahmanyam. *Sci. & Culture*, 15, 277-279 (1960); *Water Pollution Abs.*, 23, 238 (Oct. 1960).

The alkaline waste waters have a high content of organic matter and also contain chlorides, sulphates, and dyes. Ten mills at Ahmedabad are now partly neutralising by mixing the alkaline with acid waste waters and then treating with 4 cwt. of powdered gypsum per 100,000 gal., aerating, and settling. The sludge is dried on sandbeds, and the effluent discharged into sewage-treatment plant, where it is mixed with twice its volume of domestic sewage. After 12-36 hr. the mixture is neutral and can be used as fertiliser. C. O. C.

PATENT

Electrical Treatment of Water to reduce the Effects due to Hardness. A. H. Brake, G. R. Collings, and S. C. Osborne. B.P. 652,967.

The water is passed through a unit containing an electrode having knife-edge elements insulated from one another. The extreme elements are energised by a D.C. potential at 0.0001-0.001 watt dissipation per element. They are so arranged that the knife edges are the only parts of the electrode presenting bare metal to the water passing between them. Boiler-feed water fed through such a unit forms a very fine sludge and no scale. C. O. C.

Electrolytic Purification of Liquids. F. Pavelka. (XIV, p. 328.)

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Systems with Concentrated Hydrogen Peroxide. I—

The System $\text{Na}_2\text{CO}_3\text{--H}_2\text{O}_2\text{--H}_2\text{O}$. S. Z. Makarov and V. N. Chamova. *Izvestiya Akad. Nauk S.S.S.R., Otdel. Khim. Nauk*, 255-261 (May-June 1951).

The ternary system $\text{Na}_2\text{CO}_3\text{--H}_2\text{O}_2\text{--H}_2\text{O}$ was studied by the solubility method at 0, 10, and 20°C., the results being expressed on triangular phase diagrams. Stable solid phases of composition $\text{Na}_2\text{CO}_3\text{--H}_2\text{O}_2\text{--}1.5\text{H}_2\text{O}$, $\text{Na}_2\text{CO}_3\text{--H}_2\text{O}_2\text{--}2\text{H}_2\text{O}$, and $\text{Na}_2\text{CO}_3\text{--}2\text{H}_2\text{O}$ were found, but there was no evidence of the occurrence of $\text{Na}_2\text{CO}_3\text{--H}_2\text{O}_2\text{--H}_2\text{O}$, $\text{Na}_2\text{CO}_3\text{--H}_2\text{O}_2\text{--}2.5\text{H}_2\text{O}$, or $\text{Na}_2\text{CO}_3\text{--}1.5\text{H}_2\text{O}$, whose existence is indicated in the literature. The properties of the solid phases indicate them to be sodium carbonate perhydrates and not sodium percarbonate hydrates. A. E. S.

PATENTS

Sizes for Cellulose Acetate Staple Fibres. Celanese Corp. of America, M. Duke, and F. Fortess.

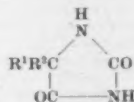
U.S.P. 2,532,406.

Warps of 20/1s count and finer can be woven satisfactorily if sized with an aq. soln. of a polyvinyl alcohol containing a deliquescent material, e.g. glycerol, sorbitol, polyethylene glycol, or urea, and having a lubricating and conditioning composition dispersed in it. C. O. C.

Hydantoin-Formaldehyde Resins—Textile Sizes, Paper Finishes, and Adhesives. du Pont and A. F. Chadwick.

U.S.P. 2,532,278.

Products of higher m.p. than those obtainable by previous methods are made by treating a hydantoin of formula—



(R^1 and $\text{R}^2 = \text{H}$ or univalent aliphatic or cycloaliphatic groups, or $\text{R}^1 = \text{H}$ and $\text{R}^2 = \text{univalent aliphatic or cycloaliphatic group}$, or R^1 and R^2 together = a bivalent aliphatic or cycloaliphatic group) with formaldehyde in presence of sufficient of an alkali-metal aliphatic mono- or di-carboxylate of < 6 C or an alkali-metal borate or phosphate to bring the mixture to pH not < 5. It is preferable that an iron deactivator, e.g. an alkali-metal phosphate, be present. C. O. C.

1:2-Diacetylenes as Bactericides, Fungicides, and Insecticides. du Pont and J. E. Kirby.

U.S.P. 2,532,558.

Compounds of formula $\text{R}^1\text{--CO--CH}=\text{CH--CO--R}^2$ ($\text{R}^1 = \text{univalent Alk, cycloalkyl, Ar, chloroaryl, or heterocyclic radical}$; $\text{R}^2 = \text{univalent Alk, cycloalkyl, Ar, or heterocyclic radical}$), e.g. 1:2-diacetylene, have good rod-, mildew-, and insect-proofing properties. C. O. C.

Fungicides. Monsanto Chemical Co. and R. O. Zerbe. B.P. 652,614.

Salts of *N*-cyanoalkyldithiocarbamic acids have excellent fungicidal properties. Thus cotton fabric treated with an aqueous dispersion of zinc ethylenbis-(*N*-β-cyanoethyl-dithiocarbamate) still retained much of its strength after an 8-week soil burial test, an untreated control being completely destroyed in 2 weeks. C. O. C.

Polymeric Polyamines. du Pont. B.P. 652,566.

The products resulting from the reductive amination of mono-olefin-carbon monoxide polymers (150-1:1) have a main carbon chain and amino nitrogen groups as lateral substituents, the N atom being directly attached to a carbon atom of the chain. They have a very wide range of uses; e.g. 5% aq. acetic acid solutions are useful as water-repellent finishes for paper and textiles and for rendering wool goods unshrinkable. They can also be used for preparing films and fibres which are readily dyed with acid dyes. They are also useful for fixing acid dyes fast to water in paper and textiles, for improving the dyeing properties of leather, as intermediates for oil-soluble dyes for colouring polyethylene, in paints, and as dispersants for metallic enamels. C. O. C.

Organosilicon isoCyanates. du Pont and D. X. Klein. U.S.P. 2,532,559.

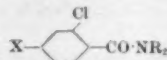
Compounds of formula $\text{R}_3\text{Si}(\text{NCO})_{1-a}$ ($\text{R} = \text{a univalent hydrocarbon radical, } a = 1-3$) can be used for water-proofing cellulosic textiles without causing tendering. The materials are impregnated with e.g. methylsilicon triisocyanate, and then cured for 5 min. at 125-175°C. C. O. C.

Polymerised Esters of Acrylic Acid—Coating and Hydrophobing Agents for Textiles and Paper. Ciba Ltd. B.P. 652,753.

Acetylene is treated with an acid and a compound containing at least one alcoholic OH group and at least one carbon chain of > 11 C or containing at least one tertiary >C-OH group. This produces esters containing the grouping >C-COOH (OR = residue of the hydroxy compound used) with or without free acrylic acid. These esters are used, as either monomers or polymers, for finishing textiles and especially to impart water-repellency. When applied as the monomers they are subsequently heat-polymerised in the fibre. C. O. C.

Insect-repelling Chlorobenzamides. J. R. Geigy A.-G. B.P. 653,027.

Compounds of formula—



($\text{X} = \text{H}$ or Cl ; $\text{R} = \text{ethyl or allyl}$) are excellent insect-repellents, of long duration and suitable for application to either textiles or the skin. C. O. C.

Polymeric Coating Compositions for use in producing Coated Fabrics. I.C.I. Ltd. and J. W. Nash. B.P. 652,737.

A coating composition which can be applied by spreading comprises a polyvinyl chloride resin, a butadiene-acrylonitrile synthetic rubber, an organic liquid which dissolves the rubber but not the resin, and a plasticiser for the resin. The coatings obtained are less thermoplastic and have greater resistance to solvents and to flex-cracking than coatings otherwise similar but based solely on polyvinyl chloride. C. O. C.

Organochlorosilanes. U.S. Secretary of Agriculture, L. A. Goldblatt, and D. M. Oldroyd. (XIII, p. 327.)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Sulphonation Reaction. XVI—Equilibrium between a Sulphonic Acid and its Acid Chloride. A. A. Spryskov and Yu. L. Kuzmina. *J. Gen. Chem. (U.S.S.R.)*, 21, 714-719 (April 1951).

When chlorosulphonic acid is used as a sulphonating agent the product is a mixture of the sulphonic acid and the sulphonyl chloride, the formation of the latter being favoured by an excess of chlorosulphonic acid, since the products are reversibly interconvertible according to the equation—



The equilibrium constant was measured for this reaction for the case in which $R = p\text{-CH}_3\text{-C}_6\text{H}_4$, and found to be 1.18 at 0°C. A state close to equilibrium was attained in 4-9 hr. after the addition of *p*-toluenesulphonyl chloride to a mixture of chlorosulphonic and sulphuric acids.

A. E. S.

Photoreaction of Pyridine in the Adsorbed State. O. V. Fialkovskaya and A. N. Terenin. *Izvestiya Akad. Nauk S.S.S.R., Otdel. Khim. Nauk*, 226-241 (May-June 1951).

Pyridine or *α*-picoline, adsorbed from the vapour under vacuum conditions on to silica gel containing AgCl, TiCl₄, or certain other salts, undergoes photoreaction in ultra-violet radiation ($\lambda < 2600 \text{ Å}$) yielding an unstable yellowish-orange product. This is considered to be a positive pyridine (*α*-picoline) ion formed by the loss of an electron. It possesses some degree of stability in absence of air and moisture if an electron acceptor, e.g. AgCl, is present. The positive pyridine ion is considered to be the precursor of the stable yellowish-brown "photo-pyridine" of Freytag, which is formed by the ultra-violet irradiation of pyridine in presence of air and moisture.

A. E. S.

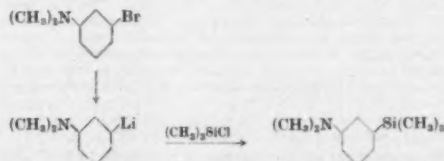
Peculiarity of Dichroism of Aromatic Molecular Compounds. R. Tsuchida, M. Kobayashi, and K. Nakamoto. *Nature*, 167, 726 (5th May 1951).

Measurements of the dichroism of the molecular compounds— $p\text{-C}_6\text{H}_4\text{O}_2$, $p\text{-C}_6\text{H}_4(\text{OH})_2$, $p\text{-C}_6\text{H}_4\text{Cl}_2$, $p\text{-C}_6\text{H}_4(\text{CH}_3)_2$, $s\text{-C}_6\text{H}_4(\text{NO}_2)_2$, $p\text{-Br-C}_6\text{H}_4\text{-NH}_2$, $s\text{-C}_6\text{H}_4\text{Cl}(\text{NO}_2)_2$, $s\text{-C}_6\text{H}_4(\text{CH}_3)_2$, and $s\text{-C}_6\text{H}_4(\text{NO}_2)_2$, $C_{10}H_{18}$ —show that the π bands are more hypsochromic and hypochromic for the light vibrating parallel to the benzene ring than for that perpendicular to the plane. This is explained as a change in electron density.

J. W. B.

Azo Dyes containing Silicon. S. V. Suntharankar and H. Gilman. *J. Org. Chem.*, 15, 1200-1210 (1950); *Chem. Abs.*, 45, 2209 (10th March 1951).

Azo dyes are made by coupling diazo and tetrazo compounds with *N,N*-dimethylaniline substituted in the *meta* position by $(\text{CH}_3)_2\text{Si-}$ or $(\text{C}_2\text{H}_5)_2\text{Si-}$, and with β -naphthol substituted in the 6-position by $(\text{CH}_3)_2\text{Si-}$ or $(\text{C}_2\text{H}_5)_2\text{Si-}$. These coupling components are made e.g. as follows—

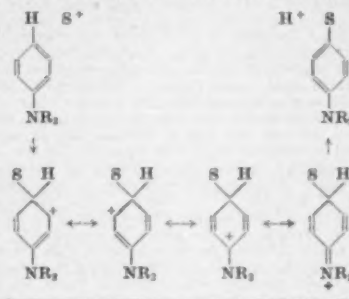


The colours produced range from the orange of the simplest members, derived from diazotised aniline, to blues given by coupling tetrazotised *o*-dianisidine with the β -naphthol derivatives. Diazo compounds couple with *p*-triphenylsilyl-*N,N*-dimethylaniline by eliminating the $(\text{C}_6\text{H}_5)_3\text{Si-}$ group. The ease of replacement of the substituents during diazo coupling was shown to decrease in the order $\text{H} > \text{R}_3\text{Si} > \text{COOH} > \text{Br}$. The failure of *m*-triphenylsilyl-*N,N*-dimethylaniline to couple with the diazonium salts from *ortho*-substituted aniline derivatives such as 2,4-dinitro-6-chloro-, 2-hydroxy-4-nitro-, and 2-trifluoromethyl-4-nitro-aniline is attributed to the steric and substituent effect of the $(\text{C}_6\text{H}_5)_3\text{Si}$ group.

E. S.

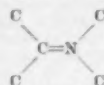
Steric Hindrance of the Auxochrome Action of the Dimethylamino Group. F. Seel and L. Suchanek. *Chem. Ber.*, 83, 438-445 (1950); *Chem. Abs.*, 45, 2451 (25th March 1951).

The reaction-promoting and *ortho*-*para*-directing action of the amino group on the electrophilic (cationoid) substitution of benzene derivatives depends upon the participation of the valency state in the activation of configuration, in which a double bond emanates from the N atom—

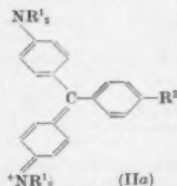


Active Configuration

(S^+ = an electrophilic substituent). This hypothesis is supported by the fact that in 2:6- $(\text{CH}_3)_2\text{C}_6\text{H}_3\text{N}(\text{CH}_3)_2$ (I) the capability of easy substitution of the *para* position in *N,N*-dimethylaniline has completely disappeared. This can be explained by increase in activation energy of the substitution: the activation configuration becomes more unstable at the *para* substitution of I because the coplanar arrangement of the atomic configuration—



favourable for formation of a double bond emanating from the N atom, is no longer possible because of the spatial hindrance of the methyl groups. Not only the directing action of the amino group, but also its action as an auxochrome depends upon the possibility that a double bond can emanate from it. This can be seen from the properties of Crystal Violet (II) and Malachite Green (III). II has three valency states of type IIa ($\text{R}^3 = \text{N}(\text{CH}_3)_2$), and III has two of type IIa ($\text{R}^3 = \text{H}$).



When the univalent cation of II is converted into the bivalent cation by increasing the acidity of the dye solution, the N atom becomes coordinatively quadrivalent by addition of a proton, and a double bond can no longer emanate from it. In this way there remain only two amino valency formulae for the bivalent cation of II, i.e. the same number and type as with III. Solutions of II containing the bivalent cation show the colour of III. Details are given of a study of how far the auxochrome action of the $\text{N}(\text{CH}_3)_2$ group is arrested by CH_3 substitution in neighbouring positions.

C. O. C.

Semi-conductivity [Photoconductivity] of Organic Dyes. II—Trypaflavine. A. T. Vartanyan. *Zhur. Fiz. Khim.*, 24, 1361-1370 (1950); *Chem. Abs.*, 45, 3709 (10th May 1951).

Chromatographically pure trypaflavine was deposited in solid films 0.1-1.0 μ , thick by crystallisation from

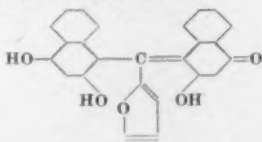
alcohol on a quartz plate. In the dark the resistance of the films was $> 10^{14}$ ohms; *in vacuo* under ultra-violet or visible radiation a photocurrent is observed which attains constant value 1 min. after irradiation starts, and decreases to zero approx. 1 min. after the light is cut off. This inertia is not due to heat emitted during illumination, as heating up to 100°C. causes no current. The relation between photocurrent and voltage is linear (up to 5000 v./cm.). The films have maxima of light absorption at 2500 and 4500 Å. The spectral distribution curves of the photocurrent depend in shape and position on the thickness of the film. For films of a few tenths of a micron, photoconduction occurs in the entire absorption band of the dye. The photocurrent divided by the energy absorbed increases with wavelength, passes through a maximum at 5000 Å., and falls to zero between 5000 and 6000 Å. Thus, when a photon is absorbed, the excited electron or hole is not free to move through the lattice; thermal energy must be supplied to achieve this. The logarithm of the photocurrent plotted against the reciprocal of the absolute temperature is linear, corresponding to an activation energy of 0.5 e.v. (at 20–100°C.). The photocurrent depends linearly on the light intensity (at 5460, 4360, and 3660 Å.). Oxygen does not affect the conductivity in the dark. Under light, the photoconductivity does not change for wavelengths > 5600 Å. With decrease in wavelength, the photoconductivity first increases owing to photo-oxidation of the dye. For light of < 4000 Å., the photocurrents *in vacuo* and in oxygen again coincide. Variation of photocurrent with oxygen pressure p (wavelength = 5520 Å.) obeys the equation—

$$i_p - i_0 = \frac{ap}{b + cp}$$

(i_p = photocurrent for p between 100 and 450 mm.; i_0 = photocurrent *in vacuo*; a , b , and c = constants). Thus the spectral distribution of the photocurrent in presence of oxygen is the result of two processes—(a) photo-oxidation of oxygen (increasing i) and (b) photo-oxidation (fading) of the dye (decreasing i). The latter process is slow, and its importance depends on the illumination and the wavelength. C. O. C.

Dye Formed by the Naphthoresorcinol Reaction of Glucuronic Acid. II. Y. Nozaki, *J. Pharm. Soc. Japan*, **64**, (7A), 9–10 (1944); III. *Ibid.*, (8A), 14–15 (1944); *Chem. Abs.*, **45**, 3599 (25th April 1951).

II—A substance having the same characteristics as the dye formed by the naphthoresorcinol (I) reaction of glucuronic acid (II) as described by Tollens (*Chem. Abs.*, **44**, 7242 (1950)) was obtained by reaction of I and II in glacial acetic acid. The products were shown to be the same and to have the structure—



III—The absorption spectrum and solubility of the pure dye are given. C. O. C.

History of Indigo. W. A. Vetterli. *Ciba Review*, (85), 3066–3071 (April 1951).

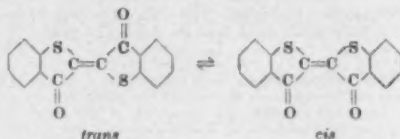
Production of [Natural] Indigo. R. Haller. *Ciba Review*, (85), 3072–3075 (April 1951). Historical. C. O. C.

Bibliography of the History of the Production and Application of Indigo. *Ciba Review*, (85), 3091 (April 1951). 33 References. C. O. C.

Relation between the Absorption Spectra and the Chemical Constitution of Dyes. XXII—*cis-trans* Isomerism in Thioindigo Dyes. G. M. Wyman and W. R. Brode. *J. Amer. Chem. Soc.*, **73**, 1487–1493 (April 1951).

Observation of the ultra-violet and visible absorption spectra of seven purified thioindigo dyes in benzene and in chloroform under several conditions of temperature and

illumination led to the conclusion that these dyes exist in solution as two forms—



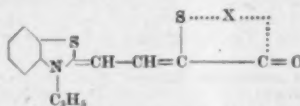
in equilibrium, the *trans* form being the more stable. The position of the equilibrium is a function of both temperature and illumination. The approximate spectral absorption curves are given for the two isomers of each dye as well as the relative amounts of each isomer present at the several equilibria. The two isomers of thioindigo and of 5:5'-dichloro-4:4':7:7'-tetramethylthioindigo were separated chromatographically and their absorption spectra obtained. Correlations between the absorption spectra and the structures of thioindigo dyes are discussed. C. O. C.

Synthesis of Trinuclear Photosensitive Dyes—I. Y. Hiko and Y. Chifu. *Rept. Sci. Research Inst. (Japan)*, **25**, 227–229 (1949); *Chem. Abs.*, **45**, 2342 (25th March 1951).

Synthesis of the following is described—6:6'-trimethoxy-1:1':1''-triethyl-10-lepidyl-4:4'-trimethinquinocyanine 1:1'-diiodide (golden-yellow crystals), 6:6'-triethoxy-1:1':1''-triethyl-10-lepidyl-4:4'-trimethinquinocyanine 1:1'-diiodide (golden-brown crystals), 6:6'-diethoxy-1:1'-dimethyl-4:4'-trimethinquinocyanine iodide (green crystals with golden-brown reflection), 6:6'-diethoxy-1:1'-diethyl-4:4'-trimethinquinocyanine iodide, and 6:6'-diethyl-4:4'-trimethinquinocyanine iodide (copper-red crystals). C. O. C.

merocyanine Dyes derived from Rhodanine. I—Properties of the Methyl-methosulphate of 2-Methylthio-5-(3'-ethylbenzthiazolylidene)-2'-ethylidene-thiazolin-4-one. Z. P. Sytkin, I. I. Levkoev, and M. V. Deichmeister. *J. Gen. Chem. (U.S.S.R.)*, **21**, 768–772 (April 1951).

When X in the formula—



is $-CS-NH-$, we have a merocyanine dye (I) that can be methylated to give a mixture of two merocyanines (II and III), for which $X = -CS-N(CH_3)-$ and $-C(S-CH_3)-N-$ respectively, the relative amounts formed depending on conditions. The further treatment of either of these with dimethyl sulphate yields the quaternary salt (IV), for which $X = -C(S-CH_3)_2-N(CH_3)_2-SO_4-CH_3-$. It has been previously observed by the authors that quaternary salts of rhodanine merocyanines are unstable, and a closer examination is now made of the decomposition of IV in aqueous soln. The ppt. that gradually forms is shown to result from the hydrolysis of IV and to be a merocyanine for which $X = -CO-N(CH_3)-$. In alcoholic soln. this gives an absorption max. at 464 mμ. (cf. 520 mμ. for I, 522 mμ. for II, 526 mμ. for III, and 536 mμ. for IV). A. E. S.

Measurement of Absorption in Solutions of Red and Yellow Dyes. III—Vegetable Dyes. A. Ringier. *Pharmazie*, **5**, 385–391 (1950); *Chem. Abs.*, **45**, 2632 (25th March 1951).

Curves and data are given for extracts of the following—*Hamatoxylon campechianum* wood, hamatoxylon, hamatin, *Casalpina echinata* wood, brasilin, *Pterocarpus santalinus* wood, frangula bark, tincture of radix rubia, cinchona bark, rotanthia root, alcaena root, *Hypericum perforatum*, *Drosera rotundifolia*, *Papaver rhoeas*, *Delphinium consolida*, *Calendula officinalis*, *Crocus sativus*, litmus, *Rottlera tinctoria*, *Garcinia hanburyi*, and berberine. C. O. C.

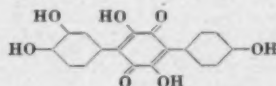
Benzalcoumaranones. E. C. Bate-Smith and T. A. Geissman. *Nature*, **167**, 688 (28th April 1951).

On the analogy of the term "flavone", applied to the paler yellow pigments containing the pyrone ring, it is

suggested that the less clumsy term "aurone" be employed in referring to the naturally occurring benzalcoumarones, which impart or contribute to flowers a golden-yellow colour.

Pigment Component of *Polyporus leucomelas*. I—Leucomelone, a Pigment. II—Synthesis of Leucomelone. M. Akagi. *J. Pharm. Soc. Japan*, **62**, 129–134, 202–206 (1942); *Chem. Abs.*, **45**, 2898 (10th April 1951).

I—The brown pigment, leucomelone, obtained from the black mushroom, *Polyporus leucomelas*, is shown to have the structure—



II—The synthesis of the above is described. C. O. C.

New Blood Pigment—Ascaricruorin. A. Treibs, H. Mendheim, and M. Lorenz. *Naturwissenschaften*, **37**, 378–379 (1950); *Chem. Abs.*, **45**, 2520 (25th March 1951).

A new blood pigment, ascaricruorin, has been obtained from the coelom fluid of the spoolworm *Ascaris lumbricoides*. It reacts like haemoglobin, but its oxygen affinity is much higher, and its CO affinity lower, than those of haemoglobin.

C. O. C.

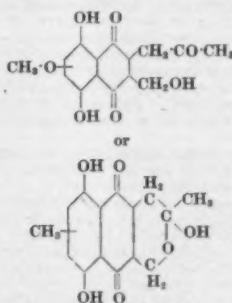
Astaxanthin as Feather Pigment. O. Volker. *Naturwissenschaften*, **37**, 309 (1950); *Chem. Abs.*, **45**, 2520 (25th March 1951).

The fire-red carotenoid pigment obtained from the feathers of *Lanius atrococcineus*, a bird from South-West Africa, has been identified as astaxanthin.

C. O. C.

Fusarubin, a Naphthaquinone Colouring Matter from *Fusaria*. H. W. Ruelius and A. Gaube. *Annalen*, **569**, 38–59 (1950); *Chem. Abs.*, **40**, 2455 (25th March 1951).

A red colouring matter, fusarubin, has been isolated from cultures of *Fusarium solani* and *F. solani* var. *Martii*. Its probable structure is—



the position of the CH₃ group remaining undetermined.

C. O. C.

Fifty Years of Lithol Red. R. L. Dittmar. *Farbe u. Lack*, **56**, 539–540 (1950); *Chem. Abs.*, **45**, 3166 (10th April 1951).

A history of the Lithol pigments.

C. O. C.

PATENTS

Polyvinylacetal Colour-formers. du Pont.

B.P. 652,928.

Alkyl Malonamates as Azo Coupling Components in Diazotype Layers. General Aniline & Film Corp. and H. C. Unkauf.

U.S.P. 2,533,185.

Compounds of formula NR¹R²-CO-CH₂-COOR³ (R¹ and R² = H, Alk, hydroxyalkyl, or aralkyl; R³ = Alk or Ar) couple with light-sensitive diazo compounds at the inter-carbonylmethylene group to form yellows of outstanding fastness to light and opacity to ultra-violet radiation.

C. O. C.

Quaternary Salts of 2-Methyl-6-methoxybenzoselenazole as Diazotype Coupling Components. General Aniline & Film Corp. and J. M. Straley.

U.S.P. 2,532,744.

Quaternary salts of heterocyclic compounds containing the grouping -C(CH₃)₂-N- in the ring are used. C. O. C.

Hair Dye. L'Oreal S.A.

B.P. 653,053.

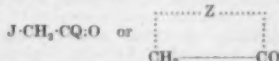
This reducing agent, e.g. thioglycolic acid, are excellent stabilisers for oxidation dyes of the phenylenediamine and aminophenol types.

C. O. C.

Oxonol Dyes. Eastman Kodak Co., L. G. S. Brooker, and S. G. Dent, Jr.

U.S.P. 2,533,206.

Oxonol dyes are prepared by condensing an organic compound of formula—



(J = cyano, carboxyl, carbalkoxyl, an acyl group of a carboxylic acid, a carbamyl, or a heterocyclic nucleus of the quinoline series; Q = OH, Alk, Ar, amino (e.g. NH₂, C₆H₄NH, etc.), carbalkoxyl, alkoxy, or a heterocyclic nucleus of the benzofuryl series; Z = atoms necessary to complete a 5- or 6-membered heterocyclic nucleus or a 5-membered carbocyclic nucleus) with a dialkoxymethyl carboxylate.

C. O. C.

Isolating the Colouring Bodies contained in Caramelised Sugar. Union Starch & Refining Co., J. E. Cleland, and J. B. Longenecker.

U.S.P. 2,533,221.

Carbon Black. Columbian Carbon Co.

B.P. 652,518.

Production from a hydrocarbon gas of carbon black of moderately fine subdivision, unique oil absorption characteristics, high degree of purity, non-acidic reaction, and good workability is described.

B.P. 652,519.

Suitable apparatus is described.

C. O. C.

Electrolytic Production of Gelatinous Titanium Hydroxide Nuclei. B. W. Allan and F. O. Rummary.

U.S.P. 2,533,208.

Acid-resistant Ultramarine. American Cyanamid Co. and F. J. Schwahl.

B.P. 653,294.

Ultramarine slurried for a few minutes with very dilute sulphuric acid, then with dilute sodium silicate, and then calcined at 300–800°C. is highly resistant to acids.

C. O. C.

Polymeric Polyamines. du Pont. (III, p. 316.)

Particle Size and Particle Shape of Pigments, and their Significance. A. Goeb. (V, this page.)

Butyl Titanate—Its Preparation, Polymerisation, and Use in Paint: Review of Present Position. G. Winter. (V, p. 320.)

Indigo Dyeing [including the Plants used] among Primitive Races. A. Bühler. (VIII, p. 323.)

Synthetic Colour-forming Binders for Photographic Emulsions. A. B. Jennings, W. A. Stanton, and J. P. Weiss. (IX, p. 323.)

Coloured Metallic Complexes of α-Oximino ketones. General Aniline & Film Corp. and D. E. Sargent. (IX, p. 324.)

V—PAINTS; ENAMELS; INKS

Particle Size and Particle Shape of Pigments, and their Significance. A. Goeb. *Farbe u. Lack*, **37**, 14–18 (1951); *Chem. Abs.*, **45**, 3165 (10th April 1951).

Pigments suitable for paints have average particle size 0.5–5.0 μ. Maximum hiding power and tinting strength are found in pigments of 0.2–0.3 μ.; below that size, undesirable effects (poor hiding power, colloidal solubility, tendency to agglomerate, high reactivity, poor flow, poor gloss) may result. Hue also varies according to particle size, e.g. Single Red I and BB. Acicular pigments also may show pleochromism, e.g. monoclinic chrome yellow, show brush marks, and give films of poor gloss. For study of the relation between hiding power and particle size a new hiding-power meter is based on measuring the light which penetrates a film of the pigment in linseed oil of a 1.47. The effect (flow of pastes) of wetting agents on various pigments is discussed. Some pigments show no effect;

some have maximum effect at one or two concentrations of wetting agents, e.g. Milori blue, ultramarine, Hansa Yellow, Peerless Black, Siegle Red 14. C. O. C.

Mechanical Properties of Red Lead and Iron Oxide Paints, and the Influence of Lead Oxide on these Properties. H. W. Talen, F. J. Hermann, and A. A. C. de Gee. *Centraal Inst. Material Onderzoek, Afdel. Verf. Circ.* No. 65, 23 pp. (1950); *Chem. Abs.*, 45, 2685 (25th March 1951).

Raw linseed oil paints containing red lead of various PbO_2 contents, Fe_2O_3 mixed with the same quantities of PbO as those contained in the red lead, or Fe_2O_3 and naphthenate driers yielded films which when aged in the case of the red lead paints increased in modulus of elasticity and tensile strength but decreased in elongation at break, Fe_2O_3 having the opposite effect. C. O. C.

Colour Stability of Clear and Opaque Titania Enamels containing Various Colour Oxides. N. K. Russell, A. L. Friedberg, and F. A. Petersen. *J. Amer. Ceram. Soc.*, 34, 28-31 (1951); *Chem. Abs.*, 45, 2644 (29th March 1951).

TiO_2 interferes with stable colour development only when it recrystallises and not when it is present only as a constituent of the glass. X-Ray data reveal no changes in crystal structures of the anatase, rutile, or colour oxides even after heating the enamels to $1500^\circ F$. C. O. C.

Butyl Titanate—Its Preparation, Polymerisation, and Use in Paint: Review of Present Position. G. Winter. *Australia, Dept. Supply, Paint Notes*, 5, 285-291 (1950); *Chem. Abs.*, 45, 4059 (10th May 1951).

Cost of production of tetrabutyl titanate (I) can be reduced by using hexane instead of a large excess of butanol when the latter reacts with $TiCl_4$ in presence of ammonia. Two molecules of I condense at $200-250^\circ C$. to a polymer (II), probably hexabutyl dititanate, splitting off 1 mol. each of butene and butanol. The reaction temperature is lowered by use of a condensing agent, e.g. an organic acid or water. II hydrolyses more slowly than I, and so gives better can stability and easier application. A typical formulation (III) is 56.9% Zn dust, 20.4% II, 0.6% high-viscosity ethyl cellulose (to prevent hard setting), and 23.1% mineral spirits. When such a formulation is painted out, II hydrolyses slowly, leaving TiO_2 , so that the resulting film is essentially inorganic. This film has excellent heat resistance and good adhesion to steel, and gives good protection against high-temperature corrosion even under humid conditions, where previous butyl titanate paints were unsatisfactory. A tendency to peel in all but very thin coats can be largely overcome by replacing Zn dust by Zn leaf. Two to three days in a ball mill are needed to grind the pigment sufficiently fine. Two coats of III followed by two coats of Al-pigmented II withstand $700^\circ C$. for long periods. Each coat must be heated to $300-500^\circ C$. before the next coat is applied. By using a light mineral oil as thinner, hot surfaces ($300^\circ C$.) may be coated. C. O. C.

PATENT

Surface Finishes of High Reflectivity and Resistance to Damp and Corrosion. J. H. Pless.

B.P. 652,766.

An undercoating containing a water-soluble silicate and a water-insoluble metal oxide, hydroxide, or salt is applied and dried, and is then coated with an aq. soln. containing calcium chloride. Either or both of the coatings may contain a resin. The undercoating may have an oil dispersed in it, and the second coating may contain $ZnCl_2$, $MgCl_2$, $Al_2(SO_4)_3$, phosphoric acid, an acid-reacting resin, a silicon alcohol or ester or a partly hydrolysed silicon ester; or alternatively the organosilicon compound can be applied as a third coating. C. O. C.

Polymeric Polyamines. du Pont. (III, p. 316.)

VI—FIBRES; YARNS; FABRICS

Mather Lecture 1951—Production and Utilisation of Textile Information. J. C. Withers. *J. Textile Inst.*, 42, p. 181-191 (May 1951).

A close survey is made of the growth of the J.T.I. and of its functions in disseminating scientific and technological information, while briefer mention is made of other relevant British and foreign journals. Textile terms and

definitions are discussed, and comments made on the inadequate supply of high-level textbooks for students. Insufficient attention is paid by most firms to the circulation of current literature, and means are suggested for remedying such omissions. J. W. B.

Micellar Structure of Native Cellulose. S. M. Mukherjee, J. Sikorski, and H. J. Woods. *Nature*, 167, 821-822 (19th May 1951).

Cotton and ramie are treated with sulphuric acid (975 g./litre) at $20^\circ C$., and with increasing severity of treatment ultimate particles 500-2500 Å. long and roughly constant in cross-section are produced. In earlier stages flat aggregates are obtained, and from a colloidal solution of these brittle transparent films isolated. X-Ray evidence indicates that crystal axial orientation persists through the aggregates, and it is suggested that the ultimate particles are themselves thinnest in the direction normal to the {101} crystallographic planes. Only a very small quantity of native cellulose particles have been obtained from jute, owing to the formation of cellulose II before the fibres disintegrate. J. W. B.

Structure and Mechanical Behaviour of Cellulose Fibres. P. Herrent and A. Lude. *Text. Research J.*, 21, 137-143 (March 1951).

The inadequacy of the simple network of single (monofilar) macromolecules attached to one another at many junctions to explain several important properties of cellulosic gels is discussed and an alternative proposal advanced. This consists of a double network, the primary part of which is an assemblage of meshes linked together by small crystals at the knots or junctions of the system. The sides of the meshes are compound instead of monofilar; they consist of bundles of macromolecular chains held together by secondary bonds. This structure makes it possible to introduce into the system the changes in internal energy hitherto lacking. An explanation is offered for the effects of degree of polymerisation and cellulose concentration on the shape of the load-extension curve and on the magnitudes of these quantities at rupture. P. C.

Cellulose-Water Relations—A Selected Bibliography with special reference to Swelling of Cotton and to its Utilisation in Water-resistant Fabrics. M. L. Rollins, H. H. Fort, and D. B. Skau. *U.S. Dept. Agriculture Library List No. 44*, 63 pp. (1949); *Chem. Abs.*, 45, 3601 (25th April 1951).

References with abstracts on swelling of cotton and other fibres, again at various R.H., mechanism of absorption of water by cellulose, application of fibre swelling in fabrics, and measurement of water-repellency. C. O. C.

Initial Stages of the Methylation of Cellulose. T. E. Timell and C. B. Purves. *Scensk Papperstidning*, 54, 303-332 (15th May 1951).

In a new method for studying in particular the earlier stages of cellulose reactions, samples taken at suitable intervals of time are nitrated as completely as possible in a non-degrading nitration mixture and then fractionated by both precipitation and solution methods. The fractions are analysed for chemical composition and degree of polymerisation to allow the distribution of the original substituents to be estimated with respect both to the different macromolecules and to the fine structure of the fibre. Applications to the methylation of cellulose with dimethyl sulphate and NaOH and to commercial alkali-soluble methyl celluloses showed that the methylation reaction begins as an intermolecular reaction, which can, however, be changed to an intramolecular one under suitable external conditions. The assumption that the accessible regions of cellulose are the principal sites of substitution led to conclusions as to the fine structure of cotton linters that agreed well with results of recent investigations. S. V. S.

Crystallinity of Celluloses after Treatment with Sodium Hydroxide (Mercerisation). P. H. Hermans and A. Weidinger. *J. Polymer Science*, 6, 533-538 (May 1951).

Both regenerated and native cellulose when treated in 18% NaOH and then washed off with boiling water show a crystallinity of 50% when measured with X-rays, which is also the figure given by rayons subject to acid hydrolysis at $100^\circ C$. and by cellulose precipitated from diluted viscose

at 100°C. Neither exclusion of oxygen during mercerisation nor addition of hydrogen peroxide to the lye has any effect on final crystallinity. C. O. C.

Auto-hydrolysis of Acidic Oxycelluloses. G. F. Davidson and H. A. Standing. *J. Textile Inst.*, **42**, τ 141- τ 144 (March 1951).

Carboxyl contents of hypobromite oxycelluloses are estimated at intervals by the Methylene Blue method, which shows a marked fall, and by the calcium acetate method, which shows only a slight reduction in carboxyl content. Washing to neutrality increases the Methylene Blue and decreases the calcium acetate results, giving better agreement and suggesting that decomposition of the oxycelluloses has occurred with production of acidic substances of low mol. wt. Similar fall in carboxyl content is produced by treating the oxycellulose in distilled water at 90°C. for 4 hr., the pH of the water falling to 3.5, indicating the formation of water-soluble acidic residues. A rise in fluidity is found at the same time. Such hydrolytic scission is shown much more effectively by periodate-chlorite oxycelluloses. It is suggested that hydrolysis of glycosidic linkages is catalysed by hydrogen ions derived from ionisation of free carboxyl groups; fission produces small units rich in carboxyl but water-soluble, thus lowering the carboxyl value. Under air-dry conditions, fission occurs at a much lower rate and leaves acidic products in the oxycellulose. J. W. B.

Oxidation of Cotton Cellulose by Nitrogen Dioxide.

T. P. Nevell. *J. Textile Inst.*, **42**, τ 91- τ 129 (March 1951).

Oxidation of cotton cellulose with nitrogen dioxide is studied over a wide range of oxygen consumption. From a stoichiometric viewpoint the production of uronic acid groups is the main reaction, but oxygen absorption indicates that side-reactions produce other types of carboxyl groups, probably in the 2:3 positions of the glucose residues. Evidence for the formation of reducing groups, probably 2:3-dialdehyde and 2:3-diketone, is provided by the alkali sensitivity of nitrogen dioxide oxycelluloses, by comparison with alginic acid and periodate oxycelluloses, by increase in weight during oxidation pointing to groups other than carboxyl, by reversible colour change on being alternately exposed to a dry and a moist atmosphere, and by the action of chlorous acid on the oxycelluloses, which increases the total carboxyl content and reduces the copper number and alkali sensitivity. Partial solubility in solutions of Methylene Blue at pH 8 makes this determination unsatisfactory, as is the determination of nitrogen content of cellulose nitrate because of partial solubility in the nitrating bath. Nitrogen dioxide oxycelluloses are more hygroscopic than unmodified cotton, and oxidation in presence of water gives similar products to those obtained under anhydrous conditions. J. W. B.

Qualitative X-Ray Study of the Oxidation of Cotton Cellulose by Nitrogen Dioxide. T. P. Nevell.

J. Textile Inst., **42**, τ 130- τ 140 (March 1951).

X-Ray powder diagrams of a series of nitrogen dioxide oxycelluloses are compared with the diagram of unmodified cotton. A high proportion of the primary alcohol groups of cellulose may be oxidised both in presence and in absence of water without destruction of the crystalline structure. Very highly oxidised materials give diffuse diagrams, and it is concluded that nitrogen dioxide is the oxidising species in the gaseous equilibrium mixtures of nitrogen dioxide and dinitrogen tetroxide employed. Powder diagrams of Methylene Blue salts of the oxycelluloses show a very strong line, also found in the diagram of Methylene Blue chloride, superimposed on the original oxycellulose diagram. Salts of oxycelluloses with other basic dyes give diagrams identical with those of the original oxycelluloses. Results are discussed in the light of Speakman and Elliott's theory of the dyeing of the crystalline phase in wool with acid dyes. J. W. B.

Polymolecularity of Viscose Rayon and Rayon Grade Pulps. W. E. Davis. *Ind. Eng. Chem.*, **43**, 516-521 (Feb. 1951).

Tyre-cord viscose rayons prepared from chemical cotton are generally superior in fatigue resistance and other dynamic properties to those made from wood pulp. By

fractionating cellulose nitrates, prepared by a non-degradative procedure from six rayons and their corresponding pulps, the proportion of low-mol. wt. material was determined, and it was found that there was fairly good correlation between fatigue resistance and the amount of low-mol. wt. material in the rayon, and between tenacity and weight-average mol. wt. There was a strong correlation between the proportion of low-mol. wt. material in the rayons and in the corresponding pulps, the chemical cottons being superior to the wood pulps in this respect. W. K. R.

Aliphatic Acid Esters of Cellulose—Preparation by Acid Chloride-Pyridine Procedure. C. J. Malm, J. W. Mench, D. L. Kendall, and G. D. Hiatt. *Ind. Eng. Chem.*, **43**, 684-688 (March 1951).

A series of cellulose esters from acetate to palmitate, of substantially complete esterification and of the same degree of polymerisation, have been prepared by the acid chloride-pyridine procedure. Reaction conditions were investigated, and the requirements for preparing satisfactory esters are described. W. K. R.

Aliphatic Acid Esters of Cellulose—Properties. C. J. Malm, J. W. Mench, D. L. Kendall, and G. D. Hiatt. *Ind. Eng. Chem.*, **43**, 688-691 (March 1951).

Data are presented on (a) m.p., (b) moisture sorption, (c) density, (d) tensile strength, (e) specific rotation, (f) refractive index, and (g) solubilities for a series of cellulose esters from acetate to palmitate, which were fully esterified and of the same degree of polymerisation. With increasing number of C atoms in the acid, (b), (c), (d), and (e) decrease. The m.p. pass through a minimum at the C_8 ester, whilst maximum solubility is reached at the C_8-C_9 esters. Refractive index varied only slightly throughout the series. W. K. R.

Composition of Bimlpatam and Mesta Fibres (*Hibiscus cannabinus*). D. B. Das, M. K. Mitra, and J. F. Wareham. *J. Textile Inst.*, **42**, τ 181- τ 184 (April 1951).

New Type of Non-woven Fabric. J. F. Ryan. *Amer. Dyestuff Rep.*, **40**, P 262-P 264 (16th April 1951).

A non-woven cotton fabric containing no bonding agent and with no interfibre fusion is described. A large degree of extensibility is coupled with high porosity and low density, and the material is suitable as a base for coating or impregnating. J. W. B.

Problems of the Structure of the Protein Molecule.

P. Jordan. *Milchwissenschaft*, **6**, 12-14 (1951); *Chem. Abs.*, **45**, 3882 (10th May 1951).

A review with emphasis on the inadequacies of current conceptions in the light of experimental evidence. The reality of long polypeptide chains is questioned. The regularities formulated by Svedberg on the basis of splitting of protein mol. offers a hopeful basis for clearer understanding of the problem. C. O. C.

Lower Resistance of Glycine Linkages in the Alkaline

Hydrolysis of Proteins. P. Desmuelle and G. Bonjour. *Compt. rend. Soc. Biol.*, **144**, 1077-1079 (1950); *Chem. Abs.*, **45**, 3881 (10th May 1951).

When proteins were treated with 5N-NaOH at 20°C. for various periods, the linkages at the amino groups of glycine were broken far more rapidly than other linkages. Silk fibroin because of its high glycine content was hydrolysed very rapidly, 50% of all its peptide linkages being broken in 24 hr. C. O. C.

Spontaneous Gelation of Alkaline Casein Dispersions.

R. L. Wormell. *Nature*, **167**, 817 (19th May 1951).

Oxidation of lactic casein and denatured peanut-protein spinning dopes, catalysed by oxidative enzymes, causes formation of disulphide cross-linkings from thiol groups and leads to irreversible gelling, which is undesirable when the dope requires stability to permit filtration and desaturation. Gelling is effectively retarded by adjusting the alkalinity with sodium sulphide instead of caustic soda. Greater sensitivity of peanut over casein protein dispersions is in accordance with the higher cystine content. J. W. B.

PATENTS

Continuous Spinning of Viscose Rayon. Courtaulds Ltd. and M. D. Rodgers. *B.P.* 652,594.

Freshly spun viscose rayon thread is passed without a break over a series of thread-advancing devices, on which

it is treated with aqueous acid at $> 65^{\circ}\text{C}$. and with an oxidising agent, e.g. hypochlorite, washed free from oxidising agent, and finally dried. The thread is not desulphurised between the aqueous acid and oxidising treatments.

W. G. C.
Spinning Viscose Rayon. Comptoir des Textiles Artificiels. B.P. 652,645.

The internal tensions in viscose rayon cakes are relaxed by causing the coagulated thread, after stretching $> 150\%$, to travel for > 3 metres during > 3 sec. before reaching the winding device and under tension such that the length of the thread remains substantially constant.

W. G. C.

Recovering Copper Oxide and a D-Tartrate from a Cuprammonium Spinning Solution. Beunitt Mills Inc. and H. Hofmann. U.S.P. 2,532,308.

Saponification of Cellulose Ester Filaments. British Celanese Ltd. B.P. 652,745.

High-tensile filamentary material of cellulose organic ester is saponified in the form of a hollow cake enclosed in a fabric sock, which passes through the hollow centre in contact with the inner surface and has its ends turned back towards and overlapping each other, thus covering all the cake surfaces. Saponification may be effected in the spinning bucket of a centrifuge, the regenerated yarn being of improved uniformity.

J. W. B.

Regenerated Cellulose Yarns with Improved Adhesion to Rubber. Dunlop Rubber Co. Ltd. and J. W. Illingworth. B.P. 653,019.

Regenerated cellulose yarns are produced by extruding a solution of an organic ester of cellulose containing a protein, e.g. casein, or a thermosetting resinous condensation product, e.g. cellulose acetate and resorcinol-formaldehyde in acetone, into a setting or gaseous evaporative medium and then completely saponifying the ester in yarn form. Such yarn has a rough surface and improved bonding to rubber, and may be used e.g. in tyre cord.

J. W. B.

Treating Casein Filaments with Mercuric Salt and Formalin to Improve their Chemical and Physical Properties. Borden Co., T. M. Buzzo, J. R. Calhoun, and J. F. Corwin. U.S.P. 2,533,356.

Casein fibre still wet from coagulation if treated with an aqueous solution of a mercuric salt and formaldehyde is not only given enhanced chemical and physical characteristics but becomes curly and wool-like.

C. O. C.

Improving the Crystallinity of Synthetic Protein Filaments. Courtaulds Ltd. and R. L. Wormell. B.P. 652,988.

Protein solution is extruded into a setting medium, and the resultant thread treated in one or more formaldehyde hardening baths, and then immersed in an aq. soln. containing a halogen-substituted aldehyde, e.g. chloral hydrate, and one or more salts, e.g. sodium sulphate, in which the protein is softened and the fibre becomes elastic, in which state it is stretched at least 50% . The product has increased birefringence and the wet and dry tenacities are improved.

J. W. B.

Improving Synthetic Fibre Crystallinity by High-frequency Sound Waves. American Viscose Corp. B.P. 652,744.

Organic polymeric material capable of being formed into fibres which show crystallinity is extruded into a liquid setting medium to form initially highly plastic fibres, which while in this state are subjected to a pulsating field of focused high-frequency sound waves, produced by an energised piezoelectric crystal and a compressional wave lens and reflector. Improved agglomeration of fibre ultimates is effected, giving greater uniformity and tenacity.

J. W. B.

Heat-stabilisation of Synthetic Linear Polyamides. I.C.I. Ltd. B.P. 652,947.

A copper salt of an organic compound, e.g. cupric acetate ($10\text{--}500$ p.p.m.), is incorporated in the polymer.

C. O. C.

Rubber Thread. Andrews-Alderfer Processing Co. Inc., S. W. Alderfer, and A. Bosshard. B.P. 653,006.

A sheet of uncured rubber stock is cut into a large number of threads, which are stretched and made to adhere to a moving surface, e.g. the upper side of an endless belt, where they are permitted to relax to remove internal stress and are cured while still on the surface.

J. W. B.

Tinsel Ribbons. Porth Textiles Ltd. and R. E. Benedict. B.P. 653,080.

Aluminium foil is slit into narrow strips, which are then interwoven with non-metallic threads. The foil may be dyed, either directly or after anodising, before or after being slit into strips.

C. O. C.

Polymeric Polyamines. du Pont. (III, p. 316.)

Silicon-containing Polymers for Coatings, Filaments, and Mouldings. du Pont and L. W. Tyran. (XIII, p. 327.)

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Detergency. I—Low-temperature Scouring of Greasy Wool. W. W. Mansfield. *Australian J. Applied Sci.*, 1, 330–347 (1950); *Chem. Abs.*, 45, 3174 (10th April 1951).

Removal of wool wax from the fibre with soap-soda solution is difficult at $< 40^{\circ}\text{C}$. because of the high viscosity of the wax. Mixtures of wax and solvent are fluid enough to be washed from wool at room temperature, addition of a little of an emulsified solvent, e.g. 1% oleic acid in benzene, to soap solution enabling wool to be scoured at about 20°C . The effects of the following on detergent efficiency are listed—solvent concentration, emulsifier concentration, temperature, age of emulsion, and nature of the solvent.

C. O. C.

Microscopic Study of Oiled Textile Fibres Immersed in Aqueous Media. D. G. Stevenson. *J. Textile Inst.*, 42, T 194–T 206 (May 1951).

Apparatus is described for observing and photographing fibres and the process of removing oil and dirt from the surface under conditions of controlled temperature and pressure allowing rapid change of surrounding liquid. Globules which form on the fibre surface under adhering oil drops are shown to be of water or aqueous solution, and are formed primarily by osmotic passage of water from the surrounding liquid to salt at or near the oil-covered fibre surface. Growth of the drop depends on the osmotic pressure and on the relative impermeabilities to water of the fibre, oil, and fibre-oil interface, and drops can also form if a moisture-saturated atmosphere surrounds the oily fibre. Water droplets may assist in detergency by pushing soil particles away from the surface. Penetration of liquid into the hollow air-filled central canal of some fibres is also studied.

J. W. B.

PATENT

Stripping Dyed Wool. Fabric Research Laboratories Inc. and E. R. Kaswell. B.P. 652,899.

More efficient stripping without alteration in the handle or strength of the fibres is obtained by immersing them in the liquid derived directly from the catholyte of an electrolytic cell containing vanadium, titanium, or chromium ions of low valency. A porous diaphragm prevents mixing of the anolyte and catholyte. Electrolysis is carried out at a current density such that the metal ions in the catholyte are reduced at least as quickly as they are oxidised to the higher valency by their action in reducing the dye on the fibre.

C. O. C.

Determination of Detergency with Artificially Soiled Fabric. G. Reutensauer and S. Dupin. (XIV, p. 328.)

VIII—DYEING

Yarn Dyeing Developments. W. Sturzacker. *Textile Weekly*, 47, 1222, 1225, 1226, 1228, 1230, 1232 (4th May 1951).

An account of the development of beam dyeing in England and of the methods and machines now in use at the author's works. The dyeing of cheeses and sliver of cotton and rayon and of loose cotton are dealt with briefly.

C. O. C.

Package Dyeing. E. L. Caswell. *Amer. Dyestuff Rep.*, 40, P 257–P 261 (10th April 1951).

Factors in the production of satisfactory dyeings on yarn in package form, e.g. kind of yarn, type of package and holder, character of winding, extraction and drying of packages, and machine characteristics, are discussed. Advantages of the 1½-in. perforated tube and the Franklin spring over the ½-in. perforated tube are stressed.

J. W. B.

Present and Future Developments in the Dyeing and Finishing of Synthetics. W. F. Brosnan. *Amer. Dyestuff Rep.*, 40, P 350-P 353 (28th May 1951).

Latest procedures in dyeing and finishing fabrics of synthetic fibres are discussed, in particular with regard to viscose, acetate rayon, nylon, and Orlon and the application of resin finishes for stabilisation and water-repellency.

J. W. B.

Effect of pH in Dyeing with Eriochrome Black T (Gy). J. Rais and O. Kryštofová. *Chem. Obzor*, 25, 136-142 (1950); *Chem. Abs.*, 45, 4044 (10th May 1951).

When dyeing with 3% Eriochrome Black T (Gy) and 2% chrome, absorption from the bath decreases with increasing pH in both the dye and the chrome baths, and the dyeing becomes bluer. A pH of 2.9-3.0 at the start of dyeing changes to 4.7-4.8 by the end of dyeing. Acidification of the bath requires slightly less acid than that corresponding to the free amino groups in the wool. The strength of the dyeing is little affected by chroming. The blue-black hue is formed if the amount of bound Cr (III) corresponds to the ratio 3 Cr : 2 mol. of dye, or is smaller. The properties of Eriochrome Black T, the acid dyeing of wool from the electrical point of view, and the chemistry of chroming are discussed.

C. O. C.

History of Indigo Dyeing [in Europe]. R. Haller. *Ciba Review*, (85), 3077-3081 (April 1951).

Indigo Dyeing [including the Plants used] among Primitive Races. A. Bühler. *Ciba Review*, (85), 3088-3091 (April 1951).

An account of the dyeing of indigo among primitive races. Although on the whole *Indigofera* plants are used, yet other plants were, and in some cases still are, used in certain areas, e.g. *Scabiosa succisa* L. and *Cheiranthus fenestralis* L. in Scandinavia, *Isatis tinctoria* L. in North Africa and India, *Polygonum tinctorium* Ait. in China and Cochín China, *Moralea tinctoria* R.B. in the East Indies, *Strobilanthes flaccidifolius* Nees in Assam and Burma, and *Sophora tinctoria* L. in Carolina and Pennsylvania.

C. O. C.

Contemporary Vat-dyeing Practice. O. W. Clark. *Amer. Dyestuff Rep.*, 40, P 315-P 322 (14th May 1951).

Continuous cloth, continuous warp, and package vat dyeing methods are reviewed, and typical dyeing recipes given.

J. W. B.

Dyeing of Algosols on Hosiery. K. A. Bridges. *Amer. Dyestuff Rep.*, 40, P 354-P 356 (28th May 1951).

Esters of leuco vat dyes are claimed to be economical and to give improved penetration and levelness, particularly in light shades, when dyeing fast colours on cotton or rayon hosiery. Working properties are discussed and application procedures given.

J. W. B.

PATENT

Dyeing Acrylonitrile Polymers with Insoluble Azo Dyes. American Viscose Corp. and R. B. Baker. U.S.P. 2,532,437.

Deep fast dyeings are produced by treating acrylonitrile polymers or copolymers in an aqueous bath containing one or more of the following—

- Benzenazo-*m*-phenylenediamine
- p*-Dimethylaminoazobenzene
- p*-Nitrobenzenazo- β -hydroxyethyl-aniline
- p*-Nitrobenzenazo- β -hydroxyethyl-ethyl-aniline
- p*-Nitrobenzenazo- β -hydroxyethyl-methyl-aniline
- p*-Nitrobenzenazo- β -hydroxyethyl-*o*-toluidine
- p*-Nitrobenzenazo-bis- β -hydroxyethyl-aniline
- p*-Nitrobenzenazodimethyl-aniline
- 2-Methoxy-4-nitrobenzenazo- β -hydroxyethyl-aniline
- 2-Methoxy-4-nitrobenzenazo- β -hydroxyethyl-methyl-aniline
- 2-Methoxy-4-nitrobenzenazo- β -hydroxyethyl-*o*-toluidine
- 2-Methoxy-4-nitrobenzenazo- β -hydroxyethyl-*m*-toluidine
- 2-Methoxy-4-nitrobenzenazodimethyl-aniline
- 2,4-Dinitrobenzenazo- β -hydroxyethyl-aniline
- 2,4-Dinitrobenzenazo- β -hydroxyethyl-ethyl-aniline
- 2,4-Dinitrobenzenazo- β -hydroxyethyl-methyl-aniline
- 2,4-Dinitrobenzenazo- β -hydroxyethyl-*o*-toluidine
- 2,4-Dinitrobenzenazo- β -hydroxyethyl-*m*-toluidine
- 2,4-Dinitrobenzenazodimethyl-aniline
- m*-Nitrobenzenazo- β -hydroxyethyl-aniline
- o*-Methoxybenzenazo- β -hydroxyethyl-aniline

2:6-Dichloro-4-nitrobenzenazo- β -hydroxyethyl-methylaniline.

The material is then dried and heated at 120-125°C. for not < 1 hr.

C. O. C.

Bibliography of the History of the Production and Application of Indigo. (IV, p. 318.)

IX—PRINTING

Reduction of Diazotised Amines on Cotton [Printing Effects]. E. R. Atkinson and A. G. Hoyle. *J. Amer. Chem. Soc.*, 73, 1841-1842 (April 1951).

3% Dyeings of C.I. 420 and 812, 4-(*p*-aminobenzenazo)-4'-nitrostilbene-2:2'-disulphonic acid, Diazo Brilliant Orange GGA Extra Concentrated CF (G), Diazo Bordeaux BACF (G), Calcomine Diazo Brilliant Green 3GD (CCC), and Rosanthrene Fast Brown RB (Ciba) were prepared on scoured bleached cotton cloth. On diazotising and boiling in water for 1 hr. they would not couple with β -naphthol. Subjecting the boiled samples to a second diazotisation still left them incapable of coupling with β -naphthol. This indicated that the original diazotisation was quantitative. Diazotised samples of the dyeings were treated for 3 min. in a cupro-ammonia reducing agent (*ibid.*, 63, 730 (1941), Method 2). After rinsing with dil. acid the samples would no longer couple with alkaline β -naphthol and their colour was slightly duller. After rediazotisation, however, they coupled with β -naphthol to produce a dyeing almost identical with that obtained on unreduced samples. As the effect produced by the reducing agent does not occur in absence of the cuproammonium ion, it is concluded that the cuproammonium ion converted the diazo compound to the original amine. This reaction can be used to obtain printing effects.

C. O. C.

Application of Indigo in Textile Printing. R. Haller. *Ciba Review*, (85), 3083-3087 (April 1951).

Historical.

C. O. C.

Synthetic Colour-forming Binders for Photographic Emulsions. A. B. Jennings, W. A. Stanton, and J. P. Weiss. *J. Soc. Motion Picture Television Engrs.*, 55, 455-476 (1950); *Chem. Abs.*, 45, 2341 (25th March 1951).

The development of synthetic colour-forming binders, particularly the acetals of polyvinyl alcohol, and their application to photographic emulsions are discussed, with special reference to du Pont Type 275 release positive colour film. These binders replace gelatin as the medium for the silver halide in the emulsion and include colour-forming substances in their structure.

C. O. C.

PATENTS

Printing Textile Fabrics with a Dye Paste comprising a Styrene-Maleic Anhydride Copolymer cross-linked with Divinylbenzene. Monsanto Chemical Co. and R. B. Seymour. U.S.P. 2,533,635.

Printing pastes, which require only small amounts of synthetic thickener, give excellent colour yields, and while being easily removed from the fabric can yet have the thickener retained as a permanent finish, consist of a dye and a 0.5-5.0% aqueous solution or dispersion of the ammonium, amine, or alkali salt of copolymerised styrene-maleic anhydride which has been cross-linked with divinylbenzene. After steaming, the thickener may be removed by washing in warm water, in which case the sodium or potassium salts of the resin are used. The ammonium and in some cases the amine salt of the resin, which become insoluble on drying, are used where the paste is desired to impart a permanent finish. The permanence of such a finish is enhanced by adding formaldehyde to the paste or by aftertreating the printed fabric with a dil. aq. soln. of a "heavy-metal" salt, e.g. the chloride, acetate, or formate of Cu, Al, Ca, or Zn.

C. O. C.

Printing with Water-soluble Dyes. Courtaulds Ltd., T. G. Allen, and A. S. Cluley. B.P. 652,985.

Addition to the printing paste of a water-insoluble cyanamide-formaldehyde condensate and a compound which on heating liberates acid, and so enables the condensate to dissolve, not only avoids bleeding of the dye during washing off but gives prints of increased fastness to washing.

C. O. C.

Colour Printing. Minister of Supply and H. Russell. B.P. 653,292.

A main printing block is made from a positive or negative obtained by exposure of the original design through a colour réseau. This main block is coloured selectively in appropriate elementary areas by several auxiliary blocks each corresponding to a separation image.

C. O. C.

Screen Printing. J. Courtney. B.P. 652,449.

The colour is applied by impregnating felt or the like with it, placing this impregnated felt on the screen, and then applying traversing pressure to the felt. This gives even application, good penetration, and quicker absorption of the colour, and enables successive sections of the fabric to be printed.

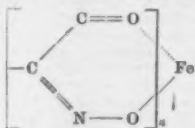
C. O. C.

Coloured Metallic Complexes of α -Oximinoketones. General Aniline & Film Corp. and D. E. Sargent.

U.S.P. 2,533,181.

U.S.P. 2,533,182.

α -Oximinoketones of structure $-\text{CO}-\text{C}(\text{N}-\text{OH})-$ or $-\text{C}(\text{OH})\text{C}(\text{NO})-$ react with certain metallic ions, e.g. ferrous ions, to yield coloured complexes which in the case of ferrous ions have the following probable structure—



($n = 2$ or 3). They can be formed *in situ* with a silver halide image by treating the latter with a compound containing a thioamide grouping which can isomerise to an isothioamide group and containing a nitrosatable methylene group adjacent to a keto group, e.g. 2-thioisobutyric acid. It is then nitrosated and treated to form a coloured metal complex. When an iron salt is used a cyan ferrous α -oximinoketone complex of exceedingly high fastness to light is produced.

C. O. C.

Bibliography of the History of the Production and Application of Indigo. (IV, p. 318.)

X—SIZING AND FINISHING

Liability to Spontaneous Combustion of Oils on Jute. A. N. Saha. *J. Textile Inst.*, 42, T 207-T 210 (May 1951).

Oxidation of linseed and mohua oils on cotton and jute, and of groundnut oil on cotton, jute, dewaxed jute, bleached jute, Cross and Bevan jute, and jute cellulose treated with 9-3% NaOH is studied using a Mackey tester. The induction period is longer, and both rate of temperature rise and final temperature are lower, for jute than for cotton. It is concluded that the smaller specific surface in comparison with cotton retards oxidation and that hemi-celluloses function as antioxidants.

J. W. B.

Interrelationship of Functional Fabric Properties resulting from the Use of Special Textile Finishes. J. F. Osterling. *Amer. Dyestuff Rep.*, 40, P 346-P 349 (28th May 1951).

Application of special textile finishes produces both primary and secondary characteristics, the first being those required, e.g. water-repellency or shrink-resistance, and the second, e.g. effect on wear-resistance or handle, being complicating effects. A list of finishing treatments is presented, with special attention to factors requiring improvement.

J. W. B.

Finishing of Face Cloths, Pile Fabrics, etc. A. I. Read. *J. Textile Inst.*, 42, P 205-P 208 (May 1951).

A brief account is given of wet and dry finishing routines for several types of face and pile fabrics.

J. W. B.

Modification of Wool by the Application of Linear Synthetic Polyamides. I—N-Methoxymethyl Polyamides. D. L. C. Jackson and M. Lipson. *Text. Research J.*, 21, 156-163 (March 1951).

Various N-methoxymethylated polyhexamethylene adipamides and unmodified nylon 66 were applied as surface deposits to wool fabric, and the effect on abrasion resistance and shrinkage was studied. N-Methoxymethylated nylons are much more effective than nylon

itself in increasing abrasion resistance but not in decreasing felting shrinkage. However, when the N-methoxymethylated nylon is first applied to wool fabric from an alcoholic solution and then hydrolysed *in situ*, the polymer confers non-felting properties on the wool. The optimum degree of substitution of the nylon for non-felting ability is 5-6% combined formaldehyde, applying 3% by wt. of polymer to the fabric. The mechanism of the action of these polymers is discussed.

P. C.

P.V.C. Leathercloth. C. A. Redfern. *Plastics Inst. Trans.*, 19, (35), 48-55 (Jan. 1951).

Polyvinyl chloride leathercloth is compared and contrasted with cellulose nitrate and drying-oil-based leathercloths and with natural leather. Methods of manufacture are described, and the merits and demerits of various constituents and treatments discussed. Consideration of its properties shows that polyvinyl chloride leathercloth is a good product which stands on its own merits and is not to be regarded merely as a substitute for other forms of leathercloth or for leather.

C. O. C.

PATENTS

Increasing the Harshness and Fullness and Decreasing the Lustre of Wool. Bigelow-Sanford Carpet Co. Inc. and S. E. Swanson. U.S.P. 2,534,314.

B.A.-type wool is rendered suitable for carpet manufacture by treating it with an aq. soln. of equal weights of aluminium chloride and sodium thiosulphate. Dyeing with neutral or direct dyes may be done before or after this treatment, but acid dyes can be applied only before the treatment.

U.S.P. 2,534,315.

Treatment with an aq. soln. of aluminium sulphate, sodium aluminium sulphate, or ammonium aluminium sulphate is used, preferably at pH 3.0-4.5.

U.S.P. 2,534,316.

The wool is treated with barium oxide, preferably by impregnating with barium hydroxide solution, and then with sulphuric acid. The treated wool can be acid-dyed.

U.S.P. 2,534,317.

The wool is placed in a bath of aq. titanyl sulphate, brought to the boil, and boiled for 5-15 min. This process may be done simultaneously with acid dyeing by adding the TiOSO_4 to the dyebath.

U.S.P. 2,534,318.

Treatment in an aqueous suspension of TiO_2 at pH 3-5 is used. It can be given by adding the TiO_2 to an acid dyebath.

U.S.P. 2,534,319.

Treatment in an acid aqueous suspension of hydrated zirconia is used. It may be combined with acid dyeing by adding 2% hydrated zirconia, previously made into an aqueous suspension, to the dyebath 15 min. before the end of dyeing.

U.S.P. 2,535,022.

Treatment with a zirconyl compound is used; if desired concurrently with dyeing, e.g. by adding 2% zirconyl sulphate to the dyebath.

C. O. C.

Electrical Treatment of Textile Fibres to decrease their Elasticity and increase their Tensile Strength. Sidney Blumenthal & Co. Inc. and L. H. V. Billiard. B.P. 652,762.

Rovings or the like are treated with a high-frequency oscillating electric field. This renders the fibres more elastic and amenable to subsequent processing, so that they can go immediately to subsequent processing without the need of waiting for them to become conditioned. They are also given permanently increased tensile strength.

C. O. C.

Non-woven Material. United Merchants & Manufacturers Inc. and L. G. Egger. U.S.P. 2,534,113.

A layer of soluble adhesive is deposited upon any suitable surface. A layer of fibres or other materials is then deposited on this adhesive, and is itself then coated with an insoluble adhesive. The composite material is then treated with a solvent to remove the first adhesive.

C. O. C.

Improving the Handle of Polymeric Linear Ester Fibres and Producing Sheer Fabrics from them. I.C.I. Ltd., J. D. H. Hall, B. P. Ridge, and J. R. Whinfield. B.P. 652,948.

Terylene and similar fibres on being treated with aq. NaOH or KOH lose weight in proportion to the length of

time they are treated, this loss in weight being uniform throughout the material. This is made use of by treating Terylene fabric in a pressure kier with 4-20% aq. caustic soda until its thickness is reduced by the desired amount.

C. O. C.

Water-repellent Finish fast to Laundering and Dry Cleaning.

du Pont and K. L. Berry.

U.S.P. 2,532,691.

Textiles and other fibrous materials are impregnated with an aq. colloidal dispersion of polytetrafluoroethylene containing as a surface-active agent a small amount of a polymeric amine salt and then baked until the amine is rendered water-insoluble.

C. O. C.

Felt-base Coverings.

D. Bruce.

B.P. 652,848.

Felt is impregnated for part of its thickness with a binder which imparts high internal strength, while the other part is treated so as to increase its tensile strength but to a lesser extent than the opposite portion. Such impregnated felt is as strong as a felt which has been uniformly impregnated and then coated with paint or linoleum composition.

C. O. C.

Coating Porous Materials.

I.C.I. Ltd., W. R. F. Gates, and R. A. Hudson.

B.P. 653,256.

A dispersion of a polymer or copolymer of a compound containing one $\text{CH}_2\text{C}=\text{C}$ group per mol. in a volatile partial solvent, e.g. polyvinyl chloride (160 parts by wt.) in tetrahydronaphthalene (29) and dioctyl phthalate (40), is coated and pressed in a layer on porous or absorbent materials, and heated in contact with a non-porous sheet to gel the dispersion and evaporate the volatile solvent, after which the non-porous sheet is stripped off. Imitation leather and wood veneer finishes, chemical-proof tank linings, and waterproof packaging material are among the products which may thus be made.

J. W. B.

Vinyl Chloride-Vinyl Acetate Copolymers as Bristles for Brushes for applying Carrotting Liquors.

W. L. Braun.

U.S.P. 2,532,991.

Vinyl chloride-vinyl acetate copolymers are remarkably resistant to the action of carrotting liquors, so that they are suitable for making bristles to withstand carrotting liquids or for impregnating or coating animal hairs or bristles for the same purpose.

C. O. C.

Heat Setting.

Fair Lawn Finishing Co. (I. p. 314.)

Organosilicon isocyanates.

du Pont and D. X. Klein. (III. p. 316.)

Crystallinity of Celluloses after Treatment with Sodium Hydroxide (Mercerisation).

P. H. Hermans and A. Weidinger. (VI. p. 320.)

Present and Future Developments in the Dyeing and Finishing of Synthetics.

W. F. Brosnan. (VIII. p. 323.)

Tanning with Mineral Salts.

T. Schachowsky. (XII. p. 326.)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Fractionation of Cellulose by means of Cuprammonium Solutions.

N. V. Shulyatikova and D. I. Mandelbaum. *J. Appl. Chem. (U.S.S.R.)*, 24, 264-273 (March 1951).

A review of methods proposed for the fractionation of cellulose according to the degree of polymerisation (D.P.) shows that they all give rise to errors and often to experimental difficulties. In many cases the cellulose is liable to suffer degradation during the determination. In particular, degradation is apt to occur in methods based on the partial dissolution of cellulose in cuprammonium solutions of low copper content. It is now found that appreciable degradation may occur even when such tests are carried out in an atmosphere of purified nitrogen, since even traces of oxygen affect the results. Satisfactory results were not obtained by the use of various antioxidants, but it was found that, even in presence of air, degradation is practically confined to the dissolved portion of the cellulose. Also, the extent to which a given wt. of cellulose dissolves in cuprammonium soln. of low copper content was found to depend not only on the concn. of copper, but also on the vol. of cuprammonium soln. taken. On the basis of these observations a new fractionation method was evolved. It consists in mixing each of a number of 1-g. samples of the same cellulose preparation with a different vol. of cupram-

monium soln. (0.25-0.26% Cu, 15.0-15.2% NH_3) at 0°C. for 2 hr. without exclusion of air. Then, in each case, the undissolved portion is separated, suitably washed, dried, and weighed; its D.P. is determined from the viscosity of its soln. in the usual cuprammonium solvent. The D.P. of the dissolved portion, which will have suffered degradation, is not measured, but is calculated on the assumption that the D.P. of the original sample is the weighted mean of the D.P. values of its components. Examination of artificial mixtures of cellulose preparations of varying D.P. shows this assumption to be justified. The fractionation technique is applied to various cellulose preparations, and the results are expressed in the form of distribution curves.

A. E. S.

Physicochemical Properties of Degraded Cellulose.

C. Pinto. *Teintex*, 16, 171-185 (April 1951).

The degradation of cellulose which takes place with acids, alkalis, and oxidising agents is discussed. Attention is paid to the yellowing of cellulose with alkalis and steam and also to various methods of estimating the degree of degradation which takes place.

B. K.

PATENTS

Papermaking Pulp from Straws, Reeds, Esparto Grass, and other Annual Growths.

Wiggins, Tespe & Co. (1919) Ltd. and S. R. H. Edge.

B.P. 652,431.

The material is boiled for 4 hr. with 0.5% NaOH soln. at pressures up to 20 lb./sq. in., drained, washed, and treated with 2-6% of NaClO_2 or ClO_2 for 2 hr. at pH 5.0 and 70°C., and the resultant pulp bleached with a hypochlorite or other bleaching agent to give a yield of e.g. about 50% bone-dry pulp from straw.

S. V. S.

Paper of High Wet Strength.

Champion Paper & Fibre Co., H. C. N. Heckel, and A. C. Salisbury.

U.S.P. 2,536,285.

When a formed web or sheet of paper is treated first with NO_2 or the dimer N_2O_4 and then with water, the fibres become bonded together with a water-resistant cement, and so high wet strength is imparted to the paper.

C. O. C.

Moisture-resistant Paper.

C. H. Child.

U.S.P. 2,539,183.

Presence in the size of a water-soluble resin of the type that gives increased wet strength enables incorporation of much more wax into the size than is normally possible and forces the wax into the paper.

C. O. C.

Coating Compositions for Paper.

D. Schoenholz and H. Terry.

U.S.P. 2,536,018.

A glossy, water- and grease-resistant coating for paper is obtained by mixing an aqueous dispersion of polyvinyl acetate (viscosity 21-100 centipoises) with an aqueous dispersion of a wax (average particle size not > 0.6 μ . in both dispersions).

C. O. C.

Pigment-coated Paper.

Dow Chemical Co., E. K. Stilbert, and A. E. Young.

U.S.P. 2,537,114.

Aqueous mineral pigment dispersions containing, as binding agent, a size, e.g. casein, and a dispersed butadiene-styrene copolymer give coatings superior to those containing a water-dispersible size as the sole binding agent.

C. O. C.

Coated Water-vapour-proof Paper.

A. Sawaro.

U.S.P. 2,538,397.

Paper coated with a mixture of coumarone-indene resin, ethyl cellulose, rosin, and polyisobutene plasticisers, paraffin wax, and zinc stearate has exceptionally low water-vapour transmission, retains its flexibility even at -20°F., withstands prolonged exposure to weather, fumes, and alcohol, and provides a satisfactory printing surface.

C. O. C.

Decorative Laminated Sheet Material.

H. J. Mallabar.

B.P. 653,531.

A process for making a coated paper for use as a surface sheet in the production of decorative laminated sheet material comprises applying a layer of white or coloured pigmented filler, e.g. precipitated barium sulphate, to paper (unfilled, if desired), brushing the coating to ensure uniform distribution, and then impregnating the coated paper with a thermosetting resin, e.g. a urea-formaldehyde or a phenol-formaldehyde resin. A backing sheet impregnated with the same or a different thermosetting resin is then bonded to the coated paper by heat and pressure.

S. V. S.

Stabilised Thermoplastic Cellulose Ether Compositions.

Hercules Powder Co., W. E. Gloor, and G. H. Pyle. *U.S.P.* 2,535,367.
Addition of a small amount of *p*-tert.-amylphenol alone or with a compound producing sulphur dioxide stabilises cellulose ether compositions to heat, light, air, weathering, etc.

Hercules Powder Co., W. Koch, and A. L. Rummelsburg. *U.S.P.* 2,535,359.
p-cyclohexylphenol is used.

Hercules Powder Co., W. Koch, and G. H. Pyle. *U.S.P.* 2,535,360.
Di-tert.-amylphenol is used.

Di-sec.-amylphenol is used. *U.S.P.* 2,535,361.

o-sec.-Amylphenol is used. *U.S.P.* 2,535,362.
Hercules Powder Co. and W. Koch.

p-sec.-Amylphenol is used. *U.S.P.* 2,535,363.
C. O. C.

Dissolution of Regenerated Cellulose with Urea-Alkali-Stannate Solvent.

United Merchants & Manufacturers Inc. and C. L. Mantell. *U.S.P.* 2,533,598.
A solvent for regenerated cellulose consists of an aqueous solution containing NaOH or KOH (8–10% by weight), urea (1–20), and sodium or potassium stannate (1.3–2.2 calc. as SnO_2). C. O. C.

Water-soluble Salts of Carboxyalkyl Celluloses.

I.C.I. Ltd., C. H. Rigby, and C. A. Macinnes. *B.P.* 652,462.

A water-soluble Na salt of a carboxyalkyl cellulose is made by incorporating into a semi-moist mixture containing cellulose the Na salt of a chlorinated aliphatic acid and NaOH in quantities that would normally leave an unconsumed excess on completion of etherification, and at least enough NaHCO_3 to convert excess NaOH to Na_2CO_3 , this NaHCO_3 being incorporated before the etherification has proceeded far. In one example coarsely disintegrated sulphite wood-pulp sheet containing 10% moisture and 82% α -cellulose (4.5 parts by wt.) is machine-mixed with ethyl alcohol (4.5) and 53% NaOH soln. (4.25) for 1 hr. at 20–25°C. To this is added finely powdered chloroacetic acid (1.81) followed by powdered NaHCO_3 (1.5), mixing is continued for 30 min. at 20–25°C., and the product, used e.g. as a thickener, is discharged into containers.

Colour-stabilisers for Cellulose Ethers.

British Celanese Ltd. *B.P.* 653,055.
Sulphites of aliphatic alcohols containing >1 OH group stabilise thermoplastic derivatives of cellulose, especially ethyl cellulose, to colour change. They have little or no effect on viscosity, heat stability, and/or resistance to crazing, and they impart no colour to the stabilised composition. C. O. C.

Organosilicon Derivatives of Cellulose.

Dow Corning Corp. and M. J. Hunter. *U.S.P.* 2,532,622.
Cellulose esters or ethers, containing on average 1–2.75 ester or ether groups per glucose unit, when treated with a compound of formula $\text{R}_3\text{ArSiHal}$ ($\text{R} = \text{Ar}$ or Alk), e.g. triphenylchlorosilane, yield derivatives containing 0.25–2.0 trialkylsilyl groups per glucose unit. The products are soluble in aromatic hydrocarbons and ethers. They are suitable for making films, mouldings, and extruded articles. C. O. C.

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Oriented Adsorption of Salts on Collagen.

A. L. Zeldes. *Doklady Akad. Nauk S.S.S.R.*, 72, 1059–1061 (1950); *Chem. Abs.*, 45, 3880 (10th May 1951).
X-Ray data are given for collagen fibres which were air-dried after being soaked in aq. soln. of CaCl_2 , KCl , Na_2SO_4 , KHCO_3 , and sodium acetate. The influence of the salts in modifying orientation of the fibres is discussed. Only CaCl_2 produces complete disorientation. C. O. C.

Tanning with Mineral Salts.

T. Schachnowskoy. *Melliand Textilber.*, 32, 302–307 (April 1951).

The structure of collagen and the processes which take

place during the weighting of silk are discussed. This is followed by a review of the reactions taking place in the tanning of leather with cobalt and other metallic salts, including the effect of neutralisation on the absorption of chrome by pelts, the acid-binding power of collagen and chrome leather, and the deposition and fixation of chrome tannins in pelts. The application of spectrophotometric measurements is indicated. B. K.

Studies in Chrome Tanning. XVII—Neutralisation of Chrome Leather prior to Colouring and Fatliquoring.

E. R. Theis, Y. Nayudamma, and C. C. Kritzing. *J. Amer. Leather Chem. Assoc.*, 46, 220–248 (April 1951).
Experimental data relating to the changes which occur when leather is washed and neutralised after chrome tanning are recorded. Ammonium bicarbonate penetrates the leather much more thoroughly and probably more quickly than sodium bicarbonate or borax, and produces a softer leather. A. S. F.

X-Ray Diffraction Studies of the Penetration of Stains and Tans into Collagen Fibrils.

O. E. A. Bolduan, T. P. Salo, and R. S. Bear. *J. Amer. Leather Chem. Assoc.*, 46, 124–138 (Feb. 1951).
Changes in the small-angle X-ray diffraction properties which occur when collagen is tanned with chromium, quebracho, and Calgon are described, and correlated where possible with previous observations. The wide-angle diffraction of dry collagen is relatively insensitive to the addition of stains or tans. A. S. F.

Yellowing of Pickled Sheepskins.

J. H. Bowes and G. O. Morse. *J. Soc. Leather Trades Chem.*, 35, 133–142 (April 1951).
The yellow discoloration which occurs when pickled sheepskins are stored is attributed partly to oxidation of the grease present but mainly to degradation of the collagen. To minimise discoloration it is suggested that the storage temperature should not exceed 30°C. A. S. F.

Hairs, Muscles, and Bacterial Flagella.

W. T. Astbury. *Nature*, 167, 880–881 (2nd June 1951).
Bacterial flagella are detached from the bacteria by shaking, isolated by centrifuging, and concentrated by ultracentrifuging. X-Ray diagrams of thin films of two specimens of flagella are similar to those of the keratin-myosin-epidermin-fibrinogen group, and on stretching show the extended β -modification. Measurements indicate that they are unimolecular hairs or muscles, and it is suggested that their rhythmic waving is produced by periodic chain folding up and down the flagellum, thus causing side-to-side movement. J. W. B.

XIII—RUBBER; RESINS; PLASTICS

Adhesion of Rubber and Textiles—Effect of Amount of Spun Staple Yarn in Textile.

E. M. Borroff, R. S. Khot, and W. C. Wake. *Ind. Eng. Chem.*, 43, 430–442 (Feb. 1951).

The adhesion of rubber to a fabric is due to the penetration of the fibres of the fabric into the rubber, rather than to penetration of rubber into the fabric. The projecting fibre ends must be pulled out or broken off to separate the rubber from the fabric, and it is shown that bond strength is a function of the number of the fibre ends protruding from the fabric and the number broken off in the rubber. Only about 10% of the available fibres are sufficiently well embedded in the rubber to fracture when the bond is broken, and this falls to a lower proportion as larger numbers of fibres are available. Better adhesion is obtained with increasing tensile strength of the fibres. The results suggest that a convenient means of obtaining good adhesion of rubber to rayon, etc. fabrics is to include a small amount of spun staple in the face to be coated, e.g. a continuous-filament warp with a suitable arrangement of picks of continuous filament and spun staple yarns. The use of sateen weaves with spun staple on the welt face enables fabrics with good adhesion characteristics to possess also the lustre of continuous-filament materials.

Limitations of Plastics.

V. E. Yarsley. *Plastics Inst. Trans.*, 19, (35), 20–37 (Jan. 1951).
Following a historical background, the limitations of plastics are considered under the headings—functional, process and processing, economic. Limitations are

admitted, but they do not lessen the intrinsic worth of plastics when used for proper purposes. C. O. C.

History of the Ethylene Chain. A. Renfrew. *Plastics Inst. Trans.*, 19, (35), 5-19 (Jan. 1951).

A historical account of the plastics which have a carbon-carbon chain skeleton, including polythene, natural and synthetic rubbers, gutta-percha, polystyrene, polyvinyl chloride, polyvinyl acetate, polymethyl methacrylate, and polymethyl chloroacrylate. C. O. C.

Thermoplastics [Ethenoid Polymers] and their Applications. A. D. Clarke. *Plastics Inst. Trans.*, 19, (35), 56-70 (Jan. 1951).

An account is given of the composition, processing, and properties of ethylene polymers, vinyl ester polymers, polyvinyl alcohol, vinylidene chloride polymers, acrylic polymers, styrene polymers, and polytetrafluoroethylene, reference being made to the various proprietary names and products. Polyvinyl chloride is discussed in detail to illustrate the influence of choice of plasticiser, stabilisers, and process upon properties and applications. C. O. C.

Latest Advances in Dry Colouring. S. E. Glick. *Modern Plastics*, 28, (6), 79-83 (1951).

Review of development of dry methods of colouring plastic moulding powders. It is suggested that this will become the most used method for producing coloured polystyrene mouldings. C. O. C.

PATENTS

Improving the Resistance of Polyethylene to Ultra-violet Radiation. Pirelli-General Cable Works Ltd., C. W. Buckles, and C. F. Williams. B.P. 652,730.

Incorporation of <1% by weight of a compound, other than a fluorescent dye, which has a strong absorption spectrum in the ultra-violet region, e.g. anthracene, improves the resistance of polyethylene to ultra-violet radiation. C. O. C.

Modified Melamine Resins. Monsanto Chemical Co. and M. J. Scott. B.P. 652,420.

Modified melamine resins having good compatibility with alkyl resins are prepared by condensing melamine, formaldehyde, and an alcohol, e.g. butanol, in presence of an arylsulphonamide (0.2-0.4 mol. per mol. of melamine), e.g. *p*-toluenesulphonamide. The products are useful in preparing surface coatings. E. C.

Flameproof Plastic Compositions. Diamond Alkali Co. B.P. 652,669.

Plastics, e.g. cellulose acetate-butyrate, rubber, polystyrene, etc., are rendered flameproof by compounding with a mixture of a highly chlorinated organic compound, e.g. chlorinated paraffin wax, a stabiliser therefor, e.g. phenyl glycidic ether or calcium stearate, and an inorganic flame retarder, e.g. antimony oxide. E. C.

Silicon-containing Polymers for Coatings, Filaments, and Mouldings. du Pont and L. W. Tyrant. U.S.P. 2,532,583.

Unsaturated alkylsilicon halides or unsaturated alkylsiliconic esters can be polymerised, with or without polymerisable carbon-chain vinyl compounds, by heating at 50°C.-b.p. in presence of a polymerisation catalyst. The polymers are soluble in common organic solvents and are suitable for coatings, filaments, and mouldings. C. O. C.

Organochlorosilanes. U.S. Secretary of Agriculture, L. A. Goldblatt, and D. M. Oldroyd. U.S.P. 2,533,240.

The addition product obtained by refluxing an olefinic terpene hydrocarbon with a halogenosilane may be used to confer water-repellency and for preparing polymers. C. O. C.

Pigmented Resins. Wingfoot Corp. and H. A. Pace. U.S.P. 2,533,196.

Any material capable of being polymerised and capable of being thickened to a pre-gel stage may be pigmented while in this stage. Uniform and stable suspension of the pigment is obtained. C. O. C.

XIV—ANALYSIS; TESTING; APPARATUS

New, Highly Specific Micro-reaction for Aluminium.

L. M. Kilberg and I. S. Mustafin. *Doklady Akad. Nauk S.S.S.R.*, 77, 285-288 (11th March 1951).

A number of α -hydroxyanthraquinone derivatives are examined as reagents for aluminium. 5:8-Dichloro-1:4-

dihydroxyanthraquinone is found to be sensitive and highly specific. The soln. under test is acidified, if necessary, and boiled with an excess of chalk to establish neutral conditions. A saturated alcoholic solution of the reagent is added (~5 drops for 1 ml. of soln.), and the soln. is again boiled, when a pink colour is produced if Al is present. The colour becomes more intense when the soln. has cooled. 5 p.p.m. of Al can be detected by the colour, and 1 p.p.m. by observing the fluorescence of the soln. in ultra-violet radiation. The reaction can be carried out as a spot-test on filter paper. Unlike the colours produced by Be, Th IV, and Cu II, the colour due to Al is unaffected by acidification of the soln. If large amounts of Fe III are present it is necessary to dilute the soln. as far as possible and to do a control test with a soln. of a ferric salt. A. E. S.

Coal Tar Colours. VIII—FD&C Yellow No. 5.

K. A. Freeman, J. H. Jones, and C. Graichen. *J. Assoc. Off. Agric. Chem.*, 33, 937-942 (1950); *Chem. Abs.*, 45, 2671 (25th March 1951).

Details of the preparation of pure FD&C Yellow No. 5 are given. At pH 5-7 the wavelength of maximum absorption of its aqueous solution is 428 ± 2 m μ . At pH 7 its aqueous solution obeys Beer's law to within $\pm 0.6\%$. The average of 17 determinations gives an extinction value of 0.0536. TiCl_3 titration is suitable for determining the pure dye content of commercial samples. C. O. C.

Optical Methods in Crystal Structure Determination.

C. A. Taylor and H. Lipsen. *Nature*, 167, 809-810 (19th May 1951).

The use of a diffraction spectrometer in which the diffraction patterns of relatively large-scale objects are observed in investigations of crystal structure is illustrated with reference to phthalocyanine. J. W. B.

Permanent Marking Ink for Porcelain Ware. M. W. Greene. *Chemist Analyst*, 39, 90 (1950); *Chem. Abs.*, 45, 2271 (25th March 1951).

Triturate a mixture of 1g. K_2CO_3 , 1g. $\text{Na}_2\text{B}_4\text{O}_7$, 2g. PbO , and 2g. dried $\text{Co}(\text{NO}_3)_2$ to a fine powder, and add 5-6g. glycerol and enough water to bring to suitable consistency. Write lightly on the ware, and heat to redness for 1-2 min.; a blue mark is imparted. C. O. C.

Oxidation of Cellulose by Chromium Trioxide—A New Method for Determining Accessibility.

R. E. Glegg. *Text. Research J.*, 21, 143-148 (March 1951).

The observation that chromium trioxide in an acetic acid-acetic anhydride mixture rapidly oxidised dry, swollen cotton linters and hardly oxidised dry, unswollen linters has been used as a basis for measuring the accessibility of cellulose. Oxidation of cellulose by this mixture takes place rapidly at first, during which the accessible portion is attacked, and then slows down suddenly. The method shows a fairly good correlation with the thallous ethoxide method over a 200-fold range of accessibility. F. C.

Testing for Artificial Fibres which are Unstable to Heat and Solvents. K. Wojatschek. *Kunststoffe u. Zellwolle*, 28, 480-482 (1950); *Chem. Abs.*, 45, 4038 (10th May 1951).

Characteristics on burning and solubility in different solvents of the following fibres are tabulated—natural and regenerated cellulose (I); cellulose acetate (II); wool, silk, and casein (III); polyvinyl (IV); polyamide (V); and polyurethane (VI) fibres. The following procedure for an unknown is recommended—Boil the sample a few min. in 5% NaOH; solubility indicates III. If insol., a fresh sample is treated with warm trichloroethylene, which dissolves IV. Solubility in cold acetic acid shows presence of II. Solubility in *m*-cresol or molten phenol denotes V and VI. These may be distinguished from each other by their solubility in cold conc. formic acid and by their odours when burnt. C. O. C.

New Microscopic Tests for the Identification of Damage to Wool and Silk. E. Kornreich. *J. Textile Inst.*, 42, T 145-T 146 (March 1951).

Tests previously described (Kornreich, *ibid.*, 41, T 321 (August 1950)) can be applied to comparatively slight damage, which is manifest by e.g. discoloration or loss

of dye, and the test has proved effective in the cases of damage by hypochlorite bleach and alkali. Examples are also described—(a) in which hair-waving lotion damaged fabric near a garment collar, the discoloured parts giving positive test results similar to those obtained by deliberate spotting; and (b) of a wool blanket, which gave a positive test result solely because of fatty acid contamination.

J. W. B.

Tentative Textile Specification No. 22, 1951—Standard Methods for Determining the Shrinkage of Knitted Fabrics and Garments containing Wool. *J. Textile Inst.*, 42, s 2-s 7 (April 1951).

Knitted wool piece goods or garments large enough to permit the cutting of test specimens, and small completed garments, e.g. socks, are tested under strictly controlled conditions (a) for relaxation shrinkage, (b) for felting in a washing machine, and (c) for felting in a milling machine of the single gravity-hammer type.

J. W. B.

Use of the Furness Milling Machine in the Determination of Shrinkage due to Felting. A. N. Davidson. *J. Textile Inst.*, 42, s 8-s 16 (April 1951).

The Furness milling machine is subjected to a rigorous examination to test its efficiency in determining felting shrinkage. Unless run at 69 or more blows per min., it behaves as a crank-driven machine, which type is believed to be unsuitable. Testing by an equal number of blows at four different speeds and loads shows that it is sensitive to both speed and load, but modification by a positive knock-off mechanism and stainless steel lining results in the same shrinkage from an equal number of blows at different speeds, although at low speeds a "between-run" error is shown. Thus modified, the machine is considered very satisfactory at speeds above 57 blows/min.

J. W. B.

Qualitative Determination of Nylon, Orlon, and Fiber V (Dacron, Terylene) in Wool Blends. H. W. Wolf. *Amer. Dyestuff Rep.*, 40, 273-276 (30th April 1951).

Wool may be separated from nylon, Orlon, and Fiber V by virtue of greater solubility in caustic alkali or sodium hypochlorite. Recoveries from known blends are tabulated with correction factors. In a mixture of all four fibres, wool is removed by sodium hypochlorite (1% available Cl), 40% caustic soda solution then removes Fiber V, and nylon is finally separated from the Orlon by dissolution in formic acid.

J. W. B.

Determination of Detergency with Artificially Soiled Fabric. G. Reutenauer and S. Dupin. *Bull. mens. I.T.E.R.G. (Inst. tech. Études Recherches Corps gras)*, 5, 84-90 (1951); *Chem. Abs.*, 45, 4066 (10th May 1951).

Artificial soils of carbon black prepared by use of oils containing unsat. fatty acids cannot be removed even by repeated washings. Use of hydrogenated oils makes it rather difficult to obtain uniform distribution of the soil. Cotton fabric (5m. x 26 cm.) is desized for 30 min. in 0.7% aq. Rapidase at 40°C., rinsed, dried, ironed, immersed for 2 min. in a mixture of 500 ml. mineral oil (S.A.E. 40), 125g. coconut oil, 2g. anthracene gas black, and 300 ml. white spirit at 110-190°C., and passed twice through a wringer. It is then washed several times in counter-current, first with previously used white spirit, then with fresh white spirit, and finally dried. Fabric so soiled was compared in the Launderometer with that treated with soils from the official Swiss Laboratory. The following differences of reflectance before and after washing were obtained with various detergents—sulphonated oleyl alcohol 17.8, 26.5; hard soap 17.6, 24.5; mixture containing 20% alkarylsulphonate, phosphate, and carboxymethyl cellulose 14.7, 21.5; secondary alkyl sulphate (20%) 14.5, 19.5; Na salt of oleylmethyltauride (30%) 14.0, 19.0; sodium oleate 13.5, 18.5; Na alkylsulphonate (40%) 12.0, 16.5; sulphonated hydroxyethylamide of palm-kernel oil acids (95%) 6.5, 12.0; K laurate 7.0, 11.5; Na₂CO₃ 5.0, 8.5; water 4.0, 5.5.

C. O. C.

Accuracy of Visual Judgments of Colour Differences on Wool Flannel. H. R. Davidson. *Amer. Dyestuff Rep.*, 40, 247-254 (16th April 1951).

Quantitative data are provided on the consistency and accuracy of colourists in describing small colour differences, on the differences between highly trained and less well trained colourists, and on the conception of "acceptable

match" of one group of colourists. Three groups of observers—highly skilled, skilled, and of no skill whatever—were selected, and 2% dyeings on wool, with small variations, made with red, blue, and green dyes. Well trained colourists are much more consistent than the remainder, and it appears that a completely untrained observer sees equally small differences but is less able to describe them. Single judgments of small differences, even by experts, are without value, while there is little agreement on the meaning of "acceptable match". J. W. B.

Retinal Colour Responses to Microstimulation. K. Motokawa, M. Ebe, Y. Arakawa, and T. Oikawa. *Nature*, 167, 729-730 (5th May 1951).

Responses to electrical stimulation of retinal receptors after exposure to light are measured and the results discussed.

J. W. B.

Colouring Matters in Soaps. T. Ruemela. *Soap (India)*, 1, (7), 10-12 (1948); *Chem. Abs.*, 45, 4065 (10th May 1951).

Colouring matters for soaps must be alkali-resistant, fast to light, sol. in hot water and soap, etc., and dermatologically harmless. Properties of the more important pigments and dyes are outlined.

C. O. C.

Colouring Copper and Copper Alloys. I—Chemical Methods. Sulphide Finishes. II—Oxide Finishes. C. Harris. *Metal Ind. (London)*, 78, 23-25, 43-44 (1951).

An account of the various methods used, including—sulphide finishes; colour "fixing" addition agents; black, steel, and fine bronzing; browns, antique and statuary patina; electrodeless immersion finishes; and electro-colouring.

C. O. C.

The Company of the Merchants of the Staple of England. B. P. Johnson. *J. Bradford Textile Soc.*, 60-75 (1949-50).

A history of the Company, England's first and greatest "regulated" Company, whose seat is at present in York.

C. O. C.

The Wool Trade and the West of England. K. G. Ponting. *J. Bradford Textile Soc.*, 83-90 (1949-50). Historical.

C. O. C.

[Colours and Colour-matching Techniques used in] Lipstick Production. *Soap, Perfumery, and Cosmetics*, 23, 1222-1228 (1950); *Chem. Abs.*, 45, 3994 (10th May 1951).

A review covering components, manufacture, colours, and colour-matching.

C. O. C.

PATENTS

Coloured Mirrors. Libbey-Owens-Ford Glass Co. B.P. 652,851. Libbey-Owens-Ford Glass Co., W. H. B.P. 652,858, Colbert, and W. L. Morgan. B.P. 652,859.

Coloured mirrors are described in which the desired colour is obtained primarily by producing interference of the light rays striking the mirrors. They have a higher reflectance than mirrors having dyes or pigments in their reflective layer and cannot break up and spot.

C. O. C.

Electrolytic Purification of Liquids. F. Pavelka. B.P. 652,442.

Apparatus comprises an anode, cathode, and two concentric diaphragms, the length of chamber between these being at least 250 times the distance between the anode and cathode diaphragms, which are arranged e.g. in the form of a helical duct, and through which liquids to be purified flow so that their particles are close to the walls.

J. W. B.

Cellulose-Water Relations—A Selected Bibliography with special reference to Swelling of Cotton and to its Utilisation in Water-resistant Fabrics. M. L. Rollins, H. H. Fort, and D. B. Skau. (VI, p. 320.)

Auto-hydrolysis of Acidic Oxycelluloses. G. F. Davidson and H. A. Standing. (VI, p. 321.)

Microscopic Study of Oiled Textile Fibres Immersed in Aqueous Media. D. G. Stevenson. (VII, p. 322.)

Fractionation of Cellulose by means of Cuprammonium Solutions. N. V. Shulyatikova and D. I. Mandelbaum. (XI, p. 325.)

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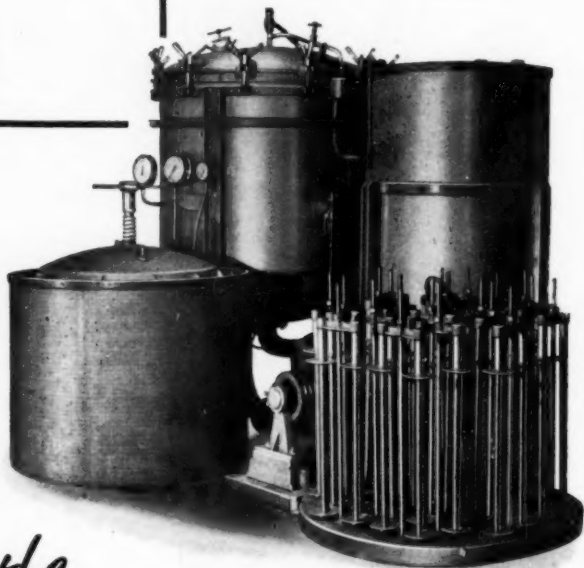
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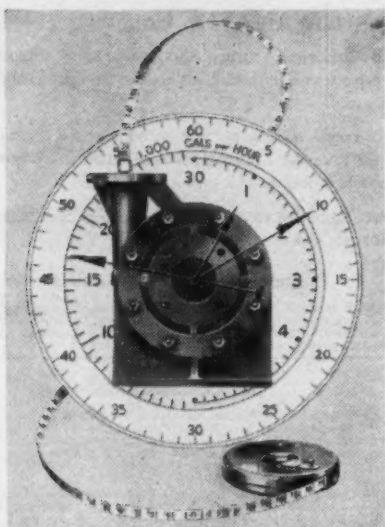
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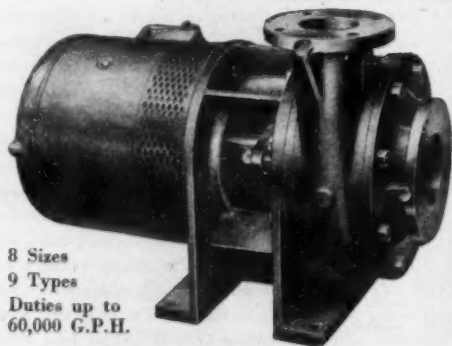


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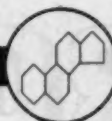
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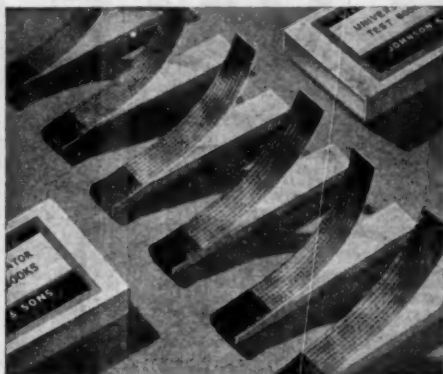
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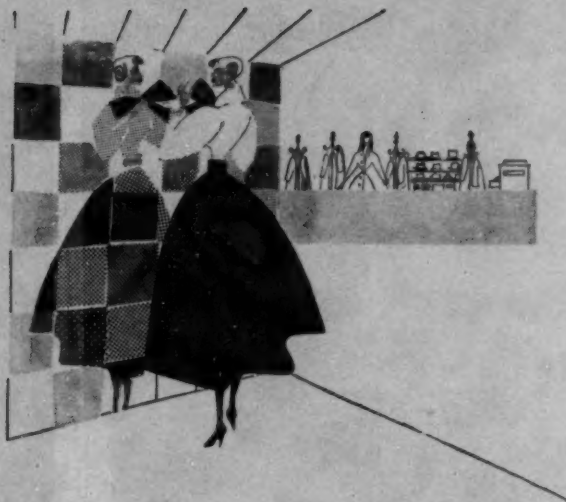
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